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CLAIMS

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[Claim(s)]

[Claim 1] Polish liquid characterized by adding the electrolyte salt which is made to distribute an abrasive material particle in an aprotic solvent, and contains the major element in the metal for polish further at least in the polish liquid for metal polish.

[Claim 2] Polish liquid according to claim 1 characterized by said electrolyte salt being the nitrate of the major element in the metal for polish, a lead sulfate, a chloride, acetate, or phosphate.

[Claim 3] Polish liquid according to claim 1 or 2 characterized by for said aprotic solvent being water and said abrasive material particle being a mono dispersion globular form particle.

[Claim 4] Polish liquid characterized by adding a water-soluble organic macromolecule in the polish liquid which made water distribute an abrasive material particle.

[Claim 5] Polish liquid according to claim 4 characterized by the concentration of said water-soluble organic macromolecule being less than [ 1wt% ].

[Claim 6] Polish liquid according to claim 4 or 5 characterized by said water-soluble organic macromolecule being a cellulose.

[Claim 7] Polish liquid given in either of claim 4 to claims 6 characterized by said abrasive material particle being a mono dispersion globular form particle.

[Claim 8] The polish approach characterized by using the polish liquid of a publication for either of claim 1 to claims 7 in the polish approach of the metal membrane formed through the insulator layer on the semi-conductor substrate.

[Claim 9] The polish approach which carries out polish clearance of the metal membrane which is in either of claim 1 to claims 7 above said insulator layer using the polish liquid of a publication in the polish approach of the metal membrane which embedded in this wiring gutter on the insulator layer in which the wiring gutter was formed beforehand, and formed membranes, and is characterized by exposing the compound front face which consists of said insulator layer and a metal membrane embedded in said wiring gutter.

[Claim 10] In the polish approach of the metal membrane which embedded in this wiring gutter on the insulator layer in which the wiring gutter was formed beforehand, and formed membranes The 1st polish process which grinds the outline of the metal membrane which is in either of claim 1 to claims 3 above said insulator layer using the polish liquid of a publication, The polish approach characterized by having the 2nd polish process which exposes the compound front face which consists of a metal membrane which ground the metal membrane which is in either of claim 4 to claims 7 above said insulator layer succeedingly using the polish liquid of a publication, and was embedded in said insulator layer and said wiring gutter.

[Claim 11] The polish approach according to claim 10 characterized by having further the 3rd process which washes said compound front face following on said 2nd polish process.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] Especially this invention relates to the polish approach at the time of forming wiring in the wiring gutter formed in the interlayer insulation film in detail by embedding a metal about the polish liquid used for the polish approach of a wiring material, and there among the wiring formation approaches of a semiconductor device, and polish liquid.

[0002]

[Description of the Prior Art] Although detailed-ization of metal wiring of aluminum etc. is needed with detailed-izing of a transistor, in the wiring formation using dry ECHINGU of the conventional metal membrane, wiring dependability degradation by the metal corrosion (metallic corrosion) by etching gas poses a problem. Then, the approach of forming embedding metal wiring in a wiring gutter or a beer hall attracts attention by forming the wiring gutter and the beer hall to substrate wiring in the interlayer insulation film beforehand, growing up a metal membrane all over an interlayer insulation film, embedding this wiring gutter and beer hall with an elevated-temperature spatter or a CVD method, and removing the metal membrane on this interlayer insulation film by selection polish of chemical machinery polish (CMP) etc. Wiring of the metal embedding mold formed of the above-mentioned process is called DAMASHIN wiring.

[0003] There is an advantage that the unification structure of wiring/beer where processing by dry etching embedded the wiring metal in the beer hall in which it was formed in self align to application of a difficult ingredient and a wiring gutter becomes possible in DAMASHIN wiring. On the other hand, in case an elasticity metal like aluminum is ground, we are anxious about the processing irregularity of the surface of metal after polish. The description of the surface of metal after such polish receives effect in the polish factor with which the property of the polish liquid (slurry) used for the polishing pressure force, a scouring pad rotational frequency, a scouring pad degree of hardness, or polish became entangled intricately.

[0004] An example of conventional CMP equipment is shown in drawing 5. Conventional CMP equipment consists of the running torque measurement section and the polish liquid feed zone of the polish head and turn table which adsorb a substrate, and a polish head. Actuation of a polish head is controlled by the torque signal from said running torque measurement section, and a polish liquid feed zone can supply alkalinity (pH> 7) and each neutral (pH=7) and acid (pH<7) polish liquid.

[0005] An example of a DAMASHIN wiring formation process which used CMP equipment for drawing 6 is shown. Hereafter, with reference to drawing 6, this process is briefly explained in order of a process.

[0006] The lower layer wiring 62 and an interlayer insulation film 63 are beforehand formed on a substrate 61, and opening of the beer hall 64 for embedding a beer plug further is carried out. Then, a metal membrane 65 (for example, aluminum) is formed the whole surface on an interlayer insulation film 63 (drawing 6 (A)). the membrane formation technique of this metal membrane 65 -- chemical vapor deposition (CVD) -- although it is possible to use law, a spatter, etc., there is the need of

embedding a metal membrane 65 in a beer hall 64 by the good membrane formation technique of embedding nature.

[0007] Next, a metal membrane 65 is ground using the polish liquid for metal polish ( drawing 6 (B) ). As polish liquid for this metal polish, an acid (about pH=3) alumina slurry etc. is used, for example. The polish rate of a metal membrane has the description that generally such an acid polish slurry is large to an interlayer insulation film. Terminal point detection of the metal membrane polish in this process is performed by carrying out the monitor of the running torque output signal from an actuation control section in the polish equipment of drawing 5 . When grinding by setting a rotational frequency constant in polish by the acid polish slurry, this will use that the running torque of a polish head goes abruptly up, if an interlayer insulation film 63 appears in a polished surface. That is, as shown in drawing 7 , running torque goes up with progress of polish, and when the level of the torque signal output set up beforehand is exceeded, a metal polish process is ended. At this event, on an interlayer insulation film 13, the metal membrane 65 remains in part as shown in drawing 6 (B) .

[0008] Then, a metal membrane 65 and an interlayer insulation film 63 are further ground using the neutral silica slurry which distributed the silica particle ( drawing 6 (C) ). This neutral silica slurry has the description of also grinding an interlayer insulation film to a metal membrane and coincidence. Consequently, flattening of the metal membrane 65 which remains on an interlayer insulation film, and an interlayer insulation film 63 advances simultaneously.

[0009] Finally polish liquid is changed from a neutral silica slurry to the alkaline silica slurry which added the potassium hydroxide etc., and polish is continued further. Since the polish rate of an interlayer insulation film has the large description to a metal membrane, this alkaline silica polish liquid can make the plug of a metal membrane 65 project from an interlayer insulation film 63 by continuing polish. Multilayer-interconnection structure is formed by performing membrane formation and patterning of the upper wiring metal next.

[0010] The conventional technique expressed above is indicated by JP,8-124886,A.

[0011]

[Problem(s) to be Solved by the Invention] The technical problem described below occurred in the conventional polish approach described above.

[0012] First, in the metal polish using conventional polish liquid, there was a problem that a deterioration layer remained to the surface of metal after polish. Although the acid slurry which distributed for example, the alumina particle is used for the conventional polish liquid for metal polish, oxidizers, such as hydrogen peroxide solution, are usually contained in this slurry, and a metal hydroxide or an oxide is formed in a surface of metal. That is, progress of metal polish is performed by forming a metal hydroxide (or oxide) deterioration layer in a surface of metal at the reaction of the oxidizer in a polish slurry, and a metal, and shaving off the deterioration layer of this front face by the scouring pad and the polish particle (here alumina particle). For this reason, the surface of metal after polish will also be inevitably covered with a metal hydroxide or an oxide. Since the deterioration layer of such a surface of metal was insulation in many cases, when it formed the upper metal wiring on the metal after polish, it had the problem that connection resistance brought about buildup. Moreover, the problem of that part separating and falling depending on the case, and the front face after polish becoming less smooth [ the hydroxide (or oxide) formed in this surface of metal ] is caused. If a local crevice exists in a metal wiring front face by such peeling, it may disconnect from the field and remarkable lowering of wiring dependability will be caused.

[0013] Moreover, in the metal polish using conventional polish liquid, there was a problem of changing polish properties, such as the polish rate, with progress of polish. It depends for the polish property of a metal membrane on the state of aggregation of the polish particle in polish liquid greatly. Since the state of aggregation of this abrasive material particle changes with the electrolytic concentration and pH in polish liquid, the component and metal in polish liquid react with progress of metal membrane polish, or if the physical properties of polish liquid change with a metal dissolving into polish liquid etc., a polish property will change at the early stages of polish, and a telophase. With conventional polish liquid, since the acid slurry for metal polish containing an oxidizer was used, the dissolution of the metal membrane

to the inside of polish liquid or its hydroxide is remarkable, and the metal ion concentration in a slurry had the inclination which increases with polish progress. Generally, if the electrolytic concentration in a slurry increases, the thickness of the electric double layer which exists in the surroundings of an abrasive material particle will decrease, and it will become easy to produce condensation of an abrasive material particle. Consequently, the technical problem that the viscosity of a polish slurry will increase with progress of polish occurred.

[0014] The above problem is nonavoidable only by only making the polish slurry for metal polish into neutrality. This is because there is a problem of the polish rate of a substrate interlayer insulation film rising on the other hand, and it becoming impossible to fully take the selection polish nature of the metal membrane to an insulator layer, although it is possible to control the dissolution to the polish slurry of the metal membrane under polish by making a polish slurry into neutrality. In this case, management of a polish process becomes very difficult.

[0015] This invention aims at improving the dependability of the semiconductor device manufactured using a polish process for the purpose of being made in order to solve the above technical problem, improving the stability of the polish property in metaled polish, and the selectivity over a substrate insulator layer, and preventing blemish generating to a surface of metal simultaneously.

[0016]

[Means for Solving the Problem] As a means to solve this technical technical problem, in the approach of grinding a metal membrane, water is made to distribute an abrasive material particle and the neutral polish liquid which added the electrolyte salt containing the metallic element which constitutes the metal membrane which serves as an object for polish further, and the polish approach using this polish liquid are offered by the 1st invention.

[0017] Moreover, in the 2nd invention, water is made to distribute an abrasive material particle and the neutral polish liquid which added the water-soluble organic macromolecule further, and the polish approach using this polish liquid are offered. As a water-soluble organic macromolecule in polish liquid, a cellulose is usable, for example and, as for the concentration, it is desirable that it is less than [ 1wt% ].

[0018] As for the abrasive material particle used in these 1st and 2nd invention, it is desirable that it is a mono dispersion globular form particle.

[0019] Moreover, in the approach of grinding the metal membrane which embedded on the insulator layer which has a wiring gutter, and was formed, this invention carries out polish clearance of the outline of a metal membrane using the polish liquid by the 1st invention first, and indicates the polish approach of continuing further and performing exposure of an insulator layer, and mirror polishing of a metal membrane using the polish liquid by the 2nd invention. You may have the process which washes the compound front face which consists of an exposed insulator layer and a metal membrane embedded in the wiring gutter further after that.

[0020] (Operation) In this invention, it grinds by dissolving the electrolyte salt containing the metal used as the object for polish, or an organic macromolecule in neutral polish liquid. Since the neutral polish liquid which is to the bases is only the thing which made pure water distribute polish particles (silica etc.) typically, deterioration layers, such as an oxide and a hydroxide, are not formed in the surface of metal to grind. In this case, polish advances by direct contact of a metal and a polish particle. In addition, strictly, although neutrality means the thing of pH=7, the neutral polish liquid as used in the field of this invention is with a pH of about six to eight polish liquid, and is for distinguishing from an about [ pH=3 ] acid conventional polish liquid, or about [ pH=9-11 ] alkaline polish liquid.

[0021] In the 1st invention, in case the metal membrane on a semi-conductor substrate is ground, it becomes possible to grind without forming in a surface of metal the metal hydroxylation film metallurgy group oxide which is an insulator by using the polish liquid which does not add an oxidizer. Moreover, it becomes possible to control change of the metal ion concentration in the polish liquid accompanying metal polish progress by dissolving electrolyte salts which serve as an object for polish beforehand, such as a metaled nitrate, a sulfate, and a chloride, in polish liquid. It decreases by this that the state of aggregation of the abrasive material particle in polish liquid changes, and it has an operation that the rate

stability of metal polish increases.

[0022] Moreover, in the 2nd invention, it becomes possible to increase the rate stability of metal polish by dissolving a water-soluble organic macromolecule in the polish liquid which does not add an oxidizer, and making the abrasive material particle condense beforehand. Furthermore, in order that selective adsorption of the water-soluble organic macromolecule may be carried out to the interlayer insulation film under a metal membrane and it may form the surface protective coat of the organic film, it also has the effectiveness of raising the polish selectivity of the metal membrane to an interlayer insulation film. The addition of this water-soluble organic macromolecule fully has that function also in minute amount addition of less than [ 1wt% ] extent in order just to form the very thin organic film in an interlayer insulation film front face.

[0023] Polish advances by direct contact to an abrasive material particle and a metal, without forming the metal oxide-film metallurgy group hydroxylation film in a metal polished surface, when the polish liquid by these 1st and 2nd invention is used. In such a case, in order to prevent blemish generating of a surface of metal, it is very effective to use the mono dispersion globular form particle which does not have caking of particles as a polish particle.

[0024] Moreover, it sets to the formation approach of DAMASHIN wiring which grinds wiring Mizogami's metal membrane formed in the interlayer insulation film, and embeds a metal membrane. Flattening polish of the parts with a main metal membrane is carried out using the polish liquid which added the electrolyte salt of a metal membrane with polish stability high as 1st polish process. The polish approach of removing the metal membrane which remains on an interlayer insulation film using the polish liquid which added the water-soluble high organic macromolecule of a selection ratio with an interlayer insulation film as 2nd polish process succeedingly is effective. The neutral penetrant remover which furthermore does not contain an abrasive material as the 3rd process may be dropped, and a washing substrate front face may be washed.

[0025]

[Embodiment of the Invention] In the gestalt of implementation of the 1st invention, it grinds in the polish liquid used for metal polish using the polish liquid which added beforehand electrolyte salts, such as a nitrate of the same metal as the metal membrane which serves as an object for polish beforehand, and a sulfate, in polish liquid. Moreover, conventionally, although oxidizers, such as hydrogen peroxide solution, are added in metal polishing, a surface of metal is oxidized or hydroxylated and this surface reaction layer was usually removed by polish, in the polish liquid of this invention, an oxidizer is not added in polish liquid, but a direct metal membrane is ground by the abrasive material in neutral polish liquid. It is easy to be the same as before [ the equipment configuration used for polish ].

[0026] By the dissolution of the above-mentioned electrolyte salt to the inside of polish liquid, it becomes possible to make an abrasive material particle condense to some extent beforehand. This controls the dissolution in the polishing liquid of the metal membrane under polish, and lifting of the viscosity of the polish liquid by metal ion concentration lifting and fluctuation of polish properties, such as a polish rate accompanying it, are avoided. Moreover, the selectivity of the metal membrane polish to an interlayer insulation film is simultaneously acquired by condensation of a polish particle. When the metal membrane which serves as an object for polish, for example is aluminum as the above-mentioned electrolyte salt, an aluminium nitrate, an aluminum sulfate, an aluminum chloride, aluminium acetate, aluminium phosphate, etc. are used. Moreover, when a metal membrane is copper, they are a copper nitrate, a copper sulfate, a copper chloride, copper acetate, copper phosphate, etc. That is, it is important to have dissolved beforehand the ion of a metal membrane used as the object for polish in polish liquid.

[0027] When the metal membrane used as the object for polish is an alloy, it is most desirable to dissolve the metal ions of all the elements that constitute the alloy into polish liquid. For example, when grinding an aluminum-Cu alloy, both aluminium nitrate and copper nitrate are beforehand dissolved into neutral polish liquid. However, in the case of an alloy which added the other type metal of a minute amount to the metal with which the metal for polish serves as a principal component, it is also good to dissolve the ion of a basis metal into neutral polish liquid. in general, when the presentation ratio of a basis metal is 95% or more, a principal component metal ion is dissolved into polish liquid -- being

sufficient. For example, what is necessary is to dissolve only the electrolyte salt containing aluminum into polish liquid, when the presentation ratio of the copper used abundantly at wiring of a semiconductor device grinds about 1% or less of aluminum-Cu alloy.

[0028] It is desirable to use a mono dispersion globular form particle without sintering of particles of a detailed globular form silica particle with a particle size of 10-300nm obtained as an abrasive material particle, for example by the solution layer depositing method (wet method) by hydrolysis of tetraethyl silicate (TEOS) etc. In this invention, in order not to add an oxidizer in polish liquid, deterioration layers, such as a hydroxide, are not formed in a surface of metal, and direct contact to an abrasive material particle and a metal arises. Under the present circumstances, generating of the blemish on the front face of a metal membrane can be substantially reduced by using a mono dispersion globular form abrasive material particle as an abrasive material particle. Except the above-mentioned detailed globular form silica particle, even if it uses oxide particles (respectively particle diameter: 10nm - about 100nm), such as aluminum, titanium, an alumina particle obtained by hydrolyzing the alkoxide of a zirconium, a titanium oxide particle, and an oxidation zirconia, the same effectiveness is acquired. In addition, although it is also possible to use the silica particle (generally called fumed silica) by the gaseous-phase pyrolysis of chlorination silicon as an abrasive material, it is desirable to grind using the mono dispersion globular form particle by the above-mentioned wet method from a viewpoint of the blemish generating prevention in the case of grinding a soft metal membrane called especially aluminum and copper.

[0029] In the gestalt of implementation of the 2nd invention, the water-soluble organic macromolecule, for example, a cellulose, is added to the abrasive material as the another technique of making the abrasive material particle condense to some extent beforehand. Organic macromolecules, such as a cellulose, stick to the front face of the silica particle which is an abrasive material particle, and have the operation which produces the chain of a silica particle in a tangle of a cellulose molecule, and polish liquid viscosity increases them as the result. In this case, a silica particle will be in the condition of having not condensed directly but having condensed indirectly by tangle of macromolecule chains, such as a cellulose.

[0030] Moreover, organic macromolecules, such as a cellulose, stick to interlayer insulation films, such as silicon oxide, and a silicon nitride, a silicon acid nitride, selectively on the substrate for polish, and do not stick to the one side metal membrane front face. For this reason, polish of a metal membrane can be advanced, an interlayer insulation film front face being covered by the organic poly membrane, and controlling polish of an interlayer insulation film. therefore -- the polish which uses this polish liquid -- a metal membrane front face -- a blemish etc. -- generating \*\*\*\*\* -- it becomes possible to grind a direct metal membrane without things, and to give the high selection ratio to a substrate interlayer insulation film. If the polish liquid which added this water-soluble organic macromolecule is used in the polish culmination in which a substrate interlayer insulation film appears, it is effective. Moreover, not only metal membranes, such as aluminum, copper, a tungsten, and titanium, but in case such effectiveness grinds conductive nitrides, such as titanium nitride and a nitriding tungsten, and metal silicide film, such as tungsten silicide and titanium silicide, it is effective.

[0031] As the water-soluble above-mentioned organic macromolecule, a glycerol, acrylic ester, etc. are [ other than a cellulose ] usable. Moreover, as for the abrasive material particle used in the gestalt of implementation of this 2nd invention, it is desirable to use mono dispersion globular form particles, such as a detailed globular form silica particle by the wet method, for the completely same reason as the case of the 1st invention.

[0032] Hereafter, the example of this invention is explained to a detail, referring to a drawing.

[0033]

[Example 1] The wiring gutter was beforehand formed in the interlayer insulation film, after that, aluminum-Cu0.5% (800nm in thickness) was embedded, membranes were formed by the elevated-temperature sputter with a substrate temperature of 450 degrees C, and the substrate for polish was formed. The pressure was set to 4mTorr(s), using Ar as sputtering gas in aluminum-Cu membrane formation. Below, the elevated-temperature sputter aluminum-Cu film formed of the above-mentioned



process is ground using the polish liquid of this invention, and the example which embedded aluminum-Cu in the wiring gutter and which embedded and formed wiring is explained.

[0034] The polish liquid used by introduction and this example is explained. The preparation odor of polish liquid prepared the neutral silica polish liquid which made pure water distribute the particle-size globular form silica particle of 10-40nm by the wet method which hydrolyzed-tetraethyl-orthochromatic silicate (TEOS) as an abrasive material particle in pure water first. Concentration of the silica particle used as an abrasive material particle was made into 10wt(s)%. The viscosity of this neutral silica polish liquid was 1.07cP(s) ( $1.07 \times 10^{-3}$ Pa-s). The polish liquid used actually was prepared by adding an electrolyte salt or an organic macromolecule in this neutral silica polish liquid.

[0035] As 1st polish liquid, the aluminium nitrate was added in the above-mentioned neutral silica polish liquid, and electrolyte addition neutral silica polish liquid was prepared. Aluminium nitrate concentration is made into 10-3 - 1wt% of within the limits, and is 0.5wt(s)% typically. Polish liquid viscosity increased to 5.09cP(s) ( $5.09 \times 10^{-3}$ Pa-s) by addition of an aluminium nitrate. That is, by adding an aluminium nitrate in neutral silica polish liquid, the silica particle was made to condense beforehand and polish liquid viscosity was enlarged about 4.5 times.

[0036] Moreover, as 2nd polish liquid, the cellulose was added in the above-mentioned neutral silica polish liquid, and macromolecule addition neutral silica polish liquid was prepared. Cellulose concentration is made into 10-6 - 10-1wt% of within the limits, and is  $4.7 \times 10^{-3}$ wt% typically. Polish liquid viscosity increased to 5.4cP(s) ( $5.40 \times 10^{-3}$ Pa-s) by addition of this cellulose. That is, by adding a cellulose in neutral silica polish liquid, the silica particle was made to condense beforehand and polish liquid viscosity was enlarged about 5 times.

[0037] The 1st of a more than and the 2nd polish liquid were used, and the substrate for polish which has the aluminum-Cu film on a front face was ground. The used polish equipment is the same as before, and uses foaming polyurethane (Rodel Nitta CO.: IC-1000 laminating pad) as a scouring pad. Moreover, the speed of supply of 35rpm and polish liquid considered rotational speed of a polish head and a turn table as a part for 60ml/.

[0038] It is drawing showing the effect of the polishing pressure force exerted on a polish rate at drawing 1. Although the polish rate increased with the increment in the polishing pressure force, the dependency of an additive kind was not seen mostly. moreover, drawing 2 -- the polishing pressure force -- 0.4kg/cm<sup>2</sup> \*\*\*\*\* -- it is drawing showing the dependency of the polish rate to the cellulose concentration at the time of grinding using the 2nd polish liquid. In experiment within the limits whose cellulose concentration is 10-6 - 10-1wt%, the polish rate is fixed at 0.2 micrometer/min, and the dependency of the polish rate to cellulose concentration is not seen. As for this, the same was said of the case of the 1st polish liquid, and the aluminium nitrate addition dependency was not accepted in a polish rate at experiment within the limits whose aluminium nitrate concentration is 10-3 - 1wt%. As mentioned above, it was checked that the aluminum film can be ground by adding an electrolyte salt and an organic macromolecule in neutral silica polish liquid beforehand, and making it condense an abrasive material particle beforehand. In addition, although the 1st of this invention and the 2nd polish liquid were improving substantially as compared with conventional polish liquid, the stability of the polish property over progress of polish, for example, the stability of a polish rate, excelled [ direction / of the 1st polish liquid which added the electrolyte salt and dissolved the metal ion into polish liquid beforehand ] in stability.

[0039] Moreover, apart from polish of the above-mentioned aluminum film, the experiment which grinds silicon oxide using the 1st of this invention and the 2nd polish liquid was conducted. Consequently, the polish rate of the silicon oxide at the time of using the 1st-polish liquid by aluminium nitrate addition was about [ in the case of the aluminum film ] about 1/10, and was about about 1/20 with the 2nd polish liquid by cellulose addition. That is, in spite of having been neutral polish liquid by using the 1st [ by this invention ], and 2nd polish liquid, it has checked that the selectivity of the aluminum polish to a substrate interlayer insulation film (here silicon oxide) was secured. However, it was checked that extent of selectivity is also excellent in the direction of the 2nd polish liquid by cellulose addition.

[0040] Drawing 3 is drawing [ polish before (immediately after membrane formation) / reflection factor / at the time of performing aluminum-Cu alloy film polish using the 1st polish liquid (aluminium nitrate addition, concentration 0.5wt%) by this invention, and the 2nd polish liquid (cellulose addition, concentration 4.7x10<sup>-2</sup>wt%) ]. In addition, polish of a metal membrane performed measurement of the reflection factor of drawing 3 in the phase (at namely, the event of the interlayer insulation film not being exposed) which advanced to the middle. Compared with the aluminum-Cu film front face immediately after elevated-temperature sputter membrane formation, even when which polish liquid is used, a reflection factor is larger than drawing 3 about a maximum of 7%. This is because the surface smoothness of the aluminum-Cu film improved by polish. When aluminium nitrate addition was compared with cellulose addition, near 300nm, compared with amyl nitrate addition neutral polish liquid, 10% or more reflection factor had the large reflection factor on the front face of aluminum which used cellulose addition neutral silica polish liquid, and it turned out that it excels by the smooth nature on the front face of aluminum after polish. In addition, even when changing cellulose concentration, in experiment [ 10-6 - 10<sup>-1</sup>wt% of ] within the limits, it was changeless to the reflection factor of a polished surface.

[0041] Thus, since oxidizers, such as hydrogen peroxide solution added by conventional polish liquid, are not added in amyl nitrate addition neutral silica polish liquid and cellulose addition neutral silica polish liquid, the metal oxide-film metallurgy group hydroxylation film is not formed during polish in a surface of metal. Moreover, since polish liquid is neutrality, a metal membrane does not dissolve in polish liquid. Furthermore, blemish generating on the front face of a metal membrane is controlled by using the very small globular form abrasive material particle.

[0042] In addition, although it was the aluminum-Cu alloy with which the metal for polish of this example contained not pure aluminium but 0.5% of copper in relation to the 1st invention, only the aluminium nitrate was used as an electrolyte salt added in the 1st polish liquid. The reason the stability of polish property sufficient also by this was acquired is because the content of the copper in an alloy is dramatically small. In general, when the content of addition metals other than the principal component in an alloy exceeds 5%, it is desirable to also double the electrolyte salt containing the addition metal, to add, and to prepare polish liquid. Of course, even when the content of the addition component metal in an alloy is 5% or less of minute amount, it cannot be overemphasized that the electrolyte salt may be added and polish liquid may be prepared.

[0043] Moreover, in embedding wiring structure which was made applicable to polish by this example, inserting barrier metal layers (for example, several nm in thickness to about several 10nm titanium etc.) between an interlayer insulation film and metal wiring is often performed. However, the thickness of such a barrier metal layer is usually dramatically thin, and since a barrier metal layer will be ground in a polish culmination just before an interlayer insulation film is exposed, fluctuation of the polish property by polish of a barrier metal layer does not usually pose a problem.

[0044]

[Example 2] In the above-mentioned example 1, even if it used any of the electrolyte salt addition polish liquid of this invention, and organic macromolecule addition polish liquid, polish of the stable metal membrane and selection polish of the metal membrane to an interlayer insulation film are possible, and it became clear that blemish generating in the front face after polish can be controlled. However, the difference in a polish property also existed between both polishes liquid, electrolyte addition polish liquid won in the stability of the polish property over progress of polish, and it became clear by one side that organic macromolecule addition polish liquid was also excellent by the selectivity over control and the interlayer insulation film of blemish generating on the front face of a metal membrane. If the above is taken into consideration, electrolyte addition polish liquid fits the activity at the flattening polish process from the early stages of polish. It is the process which this process carries out flattening of the irregularity on the front face of a metal membrane, and removes many parts of the metal membrane on an interlayer insulation film, and the stability of a polish property is searched for most. Moreover, the organic macromolecule addition abrasive material is suitable for the activity at the mirror-polishing process which removes a subsequent residual metal and exposes an interlayer insulation film. At this



process, control of the high selection ratio in polish and blemish generating to a surface of metal is called for strongly.

[0045] So, in the example 2, flattening polish which used electrolyte addition polish liquid as the 1st step is performed, mirror polishing using organic macromolecule addition polish liquid as the 2nd step is performed after that, it continues further, a washing process is performed as the 3rd step, and the example which embedded according to a series of above processes, and formed metal wiring is explained.

[0046] The outline of the polish equipment used by this example is shown in drawing 4. The polish process in this example has three steps as above-mentioned. The polish equipment of this example has three polish stages corresponding to each of this step, and while it exposes an interlayer insulation film for the flattening polish process of removing the outline of a metal membrane on the 1st polish stage 10 on the 2nd polish stage 20, it can perform a substrate washing process for the high selection mirror-polishing process which finishes a metal membrane front face on the 3rd polish stage 30. Each polish stage consists of the same configuration fundamentally, and it has the turn tables 11, 21, and 31 which equipped the top face with the scouring pad, respectively, the polish heads 12, 22, and 32, the load impression devices 13, 23, and 33, and the polish liquid feeder styles 14, 24, and 34. It can be made to be able to rotate at the rate of arbitration by the rolling mechanism, respectively, and a turn table and a polish head can apply the load of arbitration to a polish head according to a load impression device in that case. The substrates 15, 25, and 35 used as the object for polish are held by making a front face into facing down at a polish head, and perform polish or washing of a substrate by supplying polish liquid 16, 26, and 36 from a polish liquid feeder style. In addition, although the key objective of the process in the 3rd polish stage is washing of a substrate, the expression with polish liquid is used here.

[0047] In the polish equipment used by this example, the above the 1st - 3rd polish stage are arranged on one susceptor 1, and really have composition. Moreover, the substrate before polish is first installed in a loader 2, it is conveyed one by one according to the automatic conveyance device which is not illustrated after that on the 1st - the 3rd polish stage, the polish process of a three-stage is performed, and the substrate after polish / washing termination is stored in an unloader 3. Hereafter, the substrate in the polish location on each stage is distinguished as substrates 15, 25, and 35, respectively.

[0048] The substrate made applicable to polish is almost the same as the substrate used in the example 1. That is, the wiring gutter with a depth of 500nm is beforehand formed in the interlayer insulation film, and after that, by the elevated-temperature spatter with a substrate temperature of 450 degrees C, aluminum-Cu0.5% (700nm in thickness) is embedded, and membranes are formed. The membrane formation conditions of an aluminum-Cu alloy are the same as an example 1. There is surface irregularity reflecting the level difference of a substrate wiring gutter of about 300nm in this aluminum-Cu alloy film upper part.

[0049] Hereafter, the polish process in the 1st, 2nd, and 3rd polish stage in drawing 4 is explained.

[0050] The polish process in the 1st polish stage is a flattening polish process of removing the surface irregularity of an aluminum-Cu alloy film front face. the polish liquid 16 used here distributes the particle-size globular form silica particle of 40-80nm depending on the method of wet to pure water as an abrasive material particle -- making -- further -- an aluminium nitrate -- 0.5wt(s)% -- it is the added electrolyte addition polish liquid. Concentration of a silica particle was made into 10 - 20wt%. a scouring pad -- a with a degree of hardness of about (JIS-A specification and the following -- the same) 90 to 100 hard scouring pad -- using -- the load impression device 13 -- 0.3-0.4kg/cm<sup>2</sup> The high polishing pressure was impressed. The rotational frequency of a turn table 11 and the polish head 12 was set to 30 - 90rpm. This process is a process which carries out polish clearance of a lot of aluminum-Cu alloy film, and the rapidity of polish is dramatically important for it. For this reason, large polish particle and hard pad of particle size are used above a little, and it is grinding under the conditions of high voltage impression and a high-speed revolution. By the above configuration, it ground until the aluminum residual membrane was set to 100nm.

[0051] The polish process in the 2nd polish stage is a high selection mirror-polishing process which controls polish of a substrate interlayer insulation film as much as possible, and carries out mirror

polishing of the aluminum film selectively. as the polish liquid 26 of this process -- a 10nm - 40nm globular form silica particle -- 10 - 20wt% -- ~~the distributed pure water~~ -- a cellulose -- 0.1wt(s)% -- using the added organic macromolecule addition polish liquid, mirror polishing of the aluminum-Cu alloy was carried out until the substrate interlayer insulation film appeared with the scouring pad of an elasticity pad (degrees of hardness 60-90). The pressure impressed according to the load impression device 23 at this time is 0.3kg/cm<sup>2</sup>. It considered as the following low voltage force, and the rotational frequency of a turn table 21 and the polish head 22 was set to 10 - 40rpm, respectively. Here, a particle and an elasticity pad with a small particle size are used, and in order to raise the smooth nature of the polished surface in the aluminum-Cu alloy film after polish, it grinds for making selectivity of polish higher under the conditions of low voltage and a low-speed revolution. According to this process, embedding aluminum wiring (pellet SHIN aluminum wiring) without surface irregularity is obtained. [0052] The polish process in the 3rd stage is a process which washes the compound front face which was formed of the polish process to the 2nd stage, and which consists of aluminum-Cu film and an interlayer insulation film. Pure water was used as polish liquid 36 used at this process. Moreover, the impressed pressure as a scouring pad, use a with a degree of hardness of 60 or less elasticity pad, and according to the load impression device 33 is 0.3kg/cm<sup>2</sup>. It ground hereafter on the conditions of 40 or less rpm of rotational frequencies of a turn table 31 and a polish head. In addition, as polish liquid 36 used at this process, what added organic macromolecules (surfactant), such as a cellulose not more than 0.1wt%, may be used for pure water.

[0053] At a series of above polish processes, DAMASHIN wiring with which the aluminum-Cu alloy with a thickness of 500nm was embedded at the interlayer insulation film was formed. The irregularity of this aluminum-Cu wiring front face is 10nm or less, and excelling in smooth nature was checked.

[0054] In this example, as shown in drawing 4, the 1st to the 3rd made polish stage connection, and the polish equipment which can carry out automatic conveyance of the substrate between stages was used. However, even if it prepares two or more polish equipments according to individual and makes it make the 1st, 2nd, and 3rd polish process perform to each, the effectiveness of this invention is completely the same. Moreover, important processes are the 1st and 2nd polish processes, and are intrinsically [ among the above three processes ] good also by another washing means. [ of the 3rd washing process ]

[0055]

[Effect of the Invention] In the conventional metal polish, since the polish liquid which added pH modifier and the oxidizer to the dispersion liquid of a detailed abrasive material particle was used, the surface insulator layer was formed in the surface of metal, and the technical technical problem of that irregularity occurs in a surface of metal by polish, changing a polish property with progress of polish further occurred.

[0056] However, according to this invention, the above-mentioned technical problem was solvable by using the polish liquid which carried out minute amount addition of the organic macromolecules, such as an electrolyte salt which contains in neutral polish liquid the metallic element which constitutes the metal membrane for polish, or a cellulose. That is, the stability of a polish property was acquired by attaining the metal polish which was excellent in grinding a direct metal side by the abrasive material particle made to condense beforehand at smooth nature, and preventing property change of the polish liquid accompanying polish.

[0057] Moreover, in formation of embedding metal wiring, it became possible by performing flattening polish using the electrolyte salt addition abrasive material as 1st polish process, and performing the high selection mirror-polishing process using organic macromolecule addition polish liquid as 2nd polish process to form embedding wiring without surface irregularity.

[0058] The above result, formation of stable DAMASHIN wiring was attained and the remarkable effectiveness that wiring dependability improved remarkably was acquired.

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**TECHNICAL FIELD**

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[The technical field to which invention belongs] Especially this invention relates to the polish approach at the time of forming wiring in the wiring gutter formed in the interlayer insulation film in detail by embedding a metal about the polish liquid used for the polish approach of a wiring material, and there among the wiring formation approaches of a semiconductor device, and polish liquid.

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PRIOR ART

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[Description of the Prior Art] Although detailed-ization of metal wiring of aluminum etc. is needed with detailed-izing of a transistor, in the wiring formation using dry ECHINGU of the conventional metal membrane, wiring dependability degradation by the metal corrosion (metallic corrosion) by etching gas poses a problem. Then, the approach of forming embedding metal wiring in a wiring gutter or a beer hall attracts attention by forming the wiring gutter and the beer hall to substrate wiring in the interlayer insulation film beforehand, growing up a metal membrane all over an interlayer insulation film, embedding this wiring gutter and beer hall with an elevated-temperature spatter or a CVD method, and removing the metal membrane on this interlayer insulation film by selection polish of chemical machinery polish (CMP) etc. Wiring of the metal embedding mold formed of the above-mentioned process is called DAMASHIN wiring.

[0003] There is an advantage that the unification structure of wiring/beer where processing by dry etching embedded the wiring metal in the beer hall in which it was formed in self align to application of a difficult ingredient and a wiring gutter becomes possible in DAMASHIN wiring. On the other hand, in case an elasticity metal like aluminum is ground, we are anxious about the processing irregularity of the surface of metal after polish. The description of the surface of metal after such polish receives effect in the polish factor with which the property of the polish liquid (slurry) used for the polishing pressure force, a scouring pad rotational frequency, a scouring pad degree of hardness, or polish became entangled intricately.

[0004] An example of conventional CMP equipment is shown in drawing 5. Conventional CMP equipment consists of the running torque measurement section and the polish liquid feed zone of the polish head and turn table which adsorb a substrate, and a polish head. Actuation of a polish head is controlled by the torque signal from said running torque measurement section, and a polish liquid feed zone can supply alkalinity (pH> 7) and each neutral (pH=7) and acid (pH<7) polish liquid.

[0005] An example of a DAMASHIN wiring formation process which used CMP equipment for drawing 6 is shown. Hereafter, with reference to drawing 6, this process is briefly explained in order of a process.

[0006] The lower layer wiring 62 and an interlayer insulation film 63 are beforehand formed on a substrate 61, and opening of the beer hall 64 for embedding a beer plug further is carried out. Then, a metal membrane 65 (for example, aluminum) is formed the whole surface on an interlayer insulation film 63 (drawing 6 (A)). the membrane formation technique of this metal membrane 65 -- chemical vapor deposition (CVD) -- although it is possible to use law, a spatter, etc., there is the need of embedding a metal membrane 65 in a beer hall 64 by the good membrane formation technique of embedding nature.

[0007] Next, a metal membrane 65 is ground using the polish liquid for metal polish (drawing 6 (B)). As polish liquid for this metal polish, an acid (about pH=3) alumina slurry etc. is used, for example. The polish rate of a metal membrane has the description that generally such an acid polish slurry is large to an interlayer insulation film. Terminal point detection of the metal membrane polish in this process is performed by carrying out the monitor of the running torque output signal from an actuation control

section in the polish equipment of drawing 5 . When grinding by setting a rotational frequency constant in polish by the acid polish slurry, this will use that the running torque of a polish head goes abruptly up, if an interlayer insulation film 63 appears in a polished surface. That is, as shown in drawing 7 , running torque goes up with progress of polish, and when the level of the torque signal output set up beforehand is exceeded, a metal polish process is ended. At this event, on an interlayer insulation film 13, the metal membrane 65 remains in part as shown in drawing 6 (B).

[0008] Then, a metal membrane 65 and an interlayer insulation film 63 are further ground using the neutral silica slurry which distributed the silica particle- ( drawing 6 (C) ). This neutral silica slurry has the description of also grinding an interlayer insulation film to a metal membrane and coincidence. Consequently, flattening of the metal membrane 65 which remains on an interlayer insulation film, and an interlayer insulation film 63 advances simultaneously.

[0009] Finally polish liquid is changed from a neutral silica slurry to the alkaline silica slurry which added the potassium hydroxide etc., and polish is continued further. Since the polish rate of an interlayer insulation film has the large description to a metal membrane, this alkaline silica polish liquid can make the plug of a metal membrane 65 project from an interlayer insulation film 63 by continuing polish. Multilayer-interconnection structure is formed by performing membrane formation and patterning of the upper wiring metal next.

[0010] The conventional technique expressed above is indicated by JP,8-124886,A.

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EFFECT OF THE INVENTION

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[Effect of the Invention] In the conventional metal polish, since the polish liquid which added pH modifier and the oxidizer to the dispersion liquid of a detailed abrasive material particle was used, the surface insulator layer was formed in the surface of metal, and the technical technical problem of that irregularity occurs in a surface of metal by polish, changing a polish property with progress of polish further occurred.

[0056] However, according to this invention, the above-mentioned technical problem was solvable by using the polish liquid which carried out minute amount addition of the organic macromolecules, such as an electrolyte salt which contains in neutral polish liquid the metallic element which constitutes the metal membrane for polish, or a cellulose. That is, the stability of a polish property was acquired by attaining the metal polish which was excellent in grinding a direct metal side by the abrasive material particle made to condense beforehand at smooth nature, and preventing property change of the polish liquid accompanying polish.

[0057] Moreover, in formation of embedding metal wiring, it became possible by performing flattening polish using the electrolyte salt addition abrasive material as 1st polish process, and performing the high selection mirror-polishing process using organic macromolecule addition polish liquid as 2nd polish process to form embedding wiring without surface irregularity.

[0058] The above result, formation of stable DAMASHIN wiring was attained and the remarkable effectiveness that wiring dependability improved remarkably was acquired.

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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] The technical problem described below occurred in the conventional polish approach described above.

[0012] First, in the metal polish using conventional polish liquid, there was a problem that a deterioration layer remained to the surface of metal after polish. Although the acid slurry which distributed for example, the alumina particle is used for the conventional polish liquid for metal polish, oxidizers, such as hydrogen peroxide solution, are usually contained in this slurry, and a metal hydroxide or an oxide is formed in a surface of metal. That is, progress of metal polish is performed by forming a metal hydroxide (or oxide) deterioration layer in a surface of metal at the reaction of the oxidizer in a polish slurry, and a metal, and shaving off the deterioration layer of this front face by the scouring pad and the polish particle (here alumina particle). For this reason, the surface of metal after polish will also be inevitably covered with a metal hydroxide or an oxide. Since the deterioration layer of such a surface of metal was insulation in many cases, when it formed the upper metal wiring on the metal after polish, it had the problem that connection resistance brought about buildup. Moreover, the problem of that part separating and falling depending on the case, and the front face after polish becoming less smooth [ the hydroxide (or oxide) formed in this surface of metal ] is caused. If a local crevice exists in a metal wiring front face by such peeling, it may disconnect from the field and remarkable lowering of wiring dependability will be caused.

[0013] Moreover, in the metal polish using conventional polish liquid, there was a problem of changing polish properties, such as the polish rate, with progress of polish. It depends for the polish property of a metal membrane on the state of aggregation of the polish particle in polish liquid greatly. Since the state of aggregation of this abrasive material particle changes with the electrolytic concentration and pH in polish liquid, the component and metal in polish liquid react with progress of metal membrane polish, or if the physical properties of polish liquid change with a metal dissolving into polish liquid etc., a polish property will change at the early stages of polish, and a telophase. With conventional polish liquid, since the acid slurry for metal polish containing an oxidizer was used, the dissolution of the metal membrane to the inside of polish liquid or its hydroxide is remarkable, and the metal ion concentration in a slurry had the inclination which increases with polish progress. Generally, if the electrolytic concentration in a slurry increases, the thickness of the electric double layer which exists in the surroundings of an abrasive material particle will decrease, and it will become easy to produce condensation of an abrasive material particle. Consequently, the technical problem that the viscosity of a polish slurry will increase with progress of polish occurred.

[0014] The above problem is nonavoidable only by only making the polish slurry for metal polish into neutrality. This is because there is a problem of the polish rate of a substrate interlayer insulation film rising on the other hand, and it becoming impossible to fully take the selection polish nature of the metal membrane to an insulator layer, although it is possible to control the dissolution to the polish slurry of the metal membrane under polish by making a polish slurry into neutrality. In this case, management of a polish process becomes very difficult.

[0015] This invention aims at improving the dependability of the semiconductor device manufactured

using a polish process for the purpose of being made in order to solve the above technical problem, improving the stability of the polish property in metaled polish, and the selectivity over a substrate insulator layer, and preventing blemish generating to a surface of metal simultaneously.

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MEANS

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[Means for Solving the Problem] As a means to solve this technical technical problem, in the approach of grinding a metal membrane, water is made to distribute an abrasive material particle and the neutral polish liquid which added the electrolyte salt containing the metallic element which constitutes the metal membrane which serves as an object for polish further, and the polish approach using this polish liquid are offered by the 1st invention.

[0017] Moreover, in the 2nd invention, water is made to distribute an abrasive material particle and the neutral polish liquid which added the water-soluble organic macromolecule further, and the polish approach using this polish liquid are offered. As a water-soluble organic macromolecule in polish liquid, a cellulose is usable, for example and, as for the concentration, it is desirable that it is less than [ 1wt% ].

[0018] As for the abrasive material particle used in these 1st and 2nd invention, it is desirable that it is a mono dispersion globular form particle.

[0019] Moreover, in the approach of grinding the metal membrane which embedded on the insulator layer which has a wiring gutter, and was formed, this invention carries out polish clearance of the outline of a metal membrane using the polish liquid by the 1st invention first, and indicates the polish approach of continuing further and performing exposure of an insulator layer, and mirror polishing of a metal membrane using the polish liquid by the 2nd invention. You may have the process which washes the compound front face which consists of an exposed insulator layer and a metal membrane embedded in the wiring gutter further after that.

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OPERATION

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(Operation) In this invention, it grinds by dissolving the electrolyte salt containing the metal used as the object for polish, or an organic macromolecule in neutral polish liquid. Since the neutral polish liquid which is to the bases is only the thing which made pure water distribute polish particles (silica etc.) typically, deterioration layers, such as an oxide and a hydroxide, are not formed in the surface of metal to grind. In this case, polish advances by direct contact of a metal and a polish particle. In addition, strictly, although neutrality means the thing of pH=7, the neutral polish liquid as used in the field of this invention is with a pH of about six to eight polish liquid, and is for distinguishing from an about [ pH=3 ] acid conventional polish liquid, or about [ pH=9-11 ] alkaline polish liquid.

[0021] In the 1st invention, in case the metal membrane on a semi-conductor substrate is ground, it becomes possible to grind without forming in a surface of metal the metal hydroxylation film metallurgy group oxide which is an insulator by using the polish liquid which does not add an oxidizer. Moreover, it becomes possible to control change of the metal ion concentration in the polish liquid accompanying metal polish progress by dissolving electrolyte salts which serve as an object for polish beforehand, such as a metaled nitrate, a sulfate, and a chloride, in polish liquid. It decreases by this that the state of aggregation of the abrasive material particle in polish liquid changes, and it has an operation that the rate stability of metal polish increases.

[0022] Moreover, in the 2nd invention, it becomes possible to increase the rate stability of metal polish by dissolving a water-soluble organic macromolecule in the polish liquid which does not add an oxidizer, and making the abrasive material particle condense beforehand. Furthermore, in order that selective adsorption of the water-soluble organic macromolecule may be carried out to the interlayer insulation film under a metal membrane and it may form the surface protective coat of the organic film, it also has the effectiveness of raising the polish selectivity of the metal membrane to an interlayer insulation film. The addition of this water-soluble organic macromolecule fully has that function also in minute amount addition of less than [ 1wt% ] extent in order just to form the very thin organic film in an interlayer insulation film front face.

[0023] Polish advances by direct contact to an abrasive material particle and a metal, without forming the metal oxide-film metallurgy group hydroxylation film in a metal polished surface, when the polish liquid by these 1st and 2nd invention is used. In such a case, in order to prevent blemish generating of a surface of metal, it is very effective to use the mono dispersion globular form particle which does not have caking of particles as a polish particle.

[0024] Moreover, it sets to the formation approach of DAMASHIN wiring which grinds wiring Mizogami's metal membrane formed in the interlayer insulation film, and embeds a metal membrane. Flattening polish of the parts with a main metal membrane is carried out using the polish liquid which added the electrolyte salt of a metal membrane with polish stability high as 1st polish process. The polish approach of removing the metal membrane which remains on an interlayer insulation film using the polish liquid which added the water-soluble high organic macromolecule of a selection ratio with an interlayer insulation film as 2nd polish process succeedingly is effective. The neutral penetrant remover which furthermore does not contain an abrasive material as the 3rd process may be dropped, and a

washing substrate front face may be washed.

[0025]

[Embodiment of the Invention] In the gestalt of implementation of the 1st invention, it grinds in the polish liquid used for metal polish using the polish liquid which added beforehand electrolyte salts, such as a nitrate of the same metal as the metal membrane which serves as an object for polish beforehand, and a sulfate, in polish liquid. Moreover, conventionally, although oxidizers, such as hydrogen peroxide solution, are added in metal polishing, a surface of metal is oxidized or hydroxylated and this surface reaction layer was usually removed by polish, in the polish liquid of this invention, an oxidizer is not added in polish liquid, but a direct metal membrane is ground by the abrasive material in neutral polish liquid. It is easy to be the same as before [ the equipment configuration used for polish ].

[0026] By the dissolution of the above-mentioned electrolyte salt to the inside of polish liquid, it becomes possible to make an abrasive material particle condense to some extent beforehand. This controls the dissolution in the polishing liquid of the metal membrane under polish, and lifting of the viscosity of the polish liquid by metal ion concentration lifting and fluctuation of polish properties, such as a polish rate accompanying it, are avoided. Moreover, the selectivity of the metal membrane polish to an interlayer insulation film is simultaneously acquired by condensation of a polish particle. When the metal membrane which serves as an object for polish, for example is aluminum as the above-mentioned electrolyte salt, an aluminium nitrate, an aluminum sulfate, an aluminum chloride, aluminium acetate, aluminium phosphate, etc. are used. Moreover, when a metal membrane is copper, they are a copper nitrate, a copper sulfate, a copper chloride, copper acetate, copper phosphate, etc. That is, it is important to have dissolved beforehand the ion of a metal membrane used as the object for polish in polish liquid.

[0027] When the metal membrane used as the object for polish is an alloy, it is most desirable to dissolve the metal ions of all the elements that constitute the alloy into polish liquid. For example, when grinding an aluminum-Cu alloy, both aluminium nitrate and copper nitrate are beforehand dissolved into neutral polish liquid. However, in the case of an alloy which added the other type metal of a minute amount to the metal with which the metal for polish serves as a principal component, it is also good to dissolve the ion of a basis metal into neutral polish liquid. in general, when the presentation ratio of a basis metal is 95% or more, a principal component metal ion is dissolved into polish liquid -- being sufficient . For example, what is necessary is to dissolve only the electrolyte salt containing aluminum into polish liquid, when the presentation ratio of the copper used abundantly at wiring of a semiconductor device grinds about 1% or less of aluminum-Cu alloy.

[0028] It is desirable to use a mono dispersion globular form particle without sintering of particles of a detailed globular form silica particle with a particle size of 10-300nm obtained as an abrasive material particle, for example by the solution layer depositing method (wet method) by hydrolysis of tetraethyl silicate (TEOS) etc. In this invention, in order not to add an oxidizer in polish liquid, deterioration layers, such as a hydroxide, are not formed in a surface of metal, and direct contact to an abrasive material particle and a metal arises. Under the present circumstances, generating of the blemish on the front face of a metal membrane can be substantially reduced by using a mono dispersion globular form abrasive material particle as an abrasive material particle. Except the above-mentioned detailed globular form silica particle, even if it uses oxide particles (respectively particle diameter : 10nm - about 100nm), such as aluminum, titanium, an alumina particle obtained by hydrolyzing the alkoxide of a zirconium, a titanium oxide particle, and an oxidization zirconia, the same effectiveness is acquired. In addition, although it is also possible to use the silica particle (generally called fumed silica) by the gaseous-phase pyrolysis of chlorination silicon as an abrasive material, it is desirable to grind using the mono dispersion globular form particle by the above-mentioned wet method from a viewpoint of the blemish generating prevention in the case of grinding a soft metal membrane called especially aluminum and copper.

[0029] In the gestalt of implementation of the 2nd invention, the water-soluble organic macromolecule, for example, a cellulose, is added to the abrasive material as the another technique of making the abrasive material particle condense to some extent beforehand. Organic macromolecules, such as a cellulose, stick to the front face of the silica particle which is an abrasive material particle, and have the

operation which produces the chain of a silica particle in a tangle of a cellulose molecule, and polish liquid viscosity increases them as the result. In this case, a silica particle will be in the condition of having not condensed directly but having condensed indirectly by tangle of macromolecule chains, such as a cellulose.

[0030] Moreover, organic macromolecules, such as a cellulose, stick to interlayer insulation films, such as silicon oxide, and a silicon nitride, a silicon acid nitride, selectively on the substrate for polish, and do not stick to the one side metal membrane front face. For this reason, polish of a metal membrane can be advanced, an interlayer insulation film front face being covered by the organic poly membrane, and controlling polish of an interlayer insulation film. therefore -- the polish which uses this polish liquid -- a metal membrane front face -- a blemish etc. -- generating \*\*\*\*\* -- it becomes possible to grind a direct metal membrane without things, and to give the high selection ratio to a substrate interlayer insulation film. If the polish liquid which added this water-soluble organic macromolecule is used in the polish culmination in which a substrate interlayer insulation film appears, it is effective. Moreover, not only metal membranes, such as aluminum, copper, a tungsten, and titanium, but in case such effectiveness grinds conductive nitrides, such as titanium nitride and a nitriding tungsten, and metal silicide film, such as tungsten silicide and titanium silicide, it is effective.

[0031] As the water-soluble above-mentioned organic macromolecule, a glycerol, acrylic ester, etc. are [ other than a cellulose ] usable. Moreover, as for the abrasive material particle used in the gestalt of implementation of this 2nd invention, it is desirable to use mono dispersion globular form particles, such as a detailed globular form silica particle by the wet method, for the completely same reason as the case of the 1st invention.

[0032] Hereafter, the example of this invention is explained to a detail, referring to a drawing.

[0033]

[Example 1] The wiring gutter was beforehand formed in the interlayer insulation film, after that, aluminum-Cu0.5% (800nm in thickness) was embedded, membranes were formed by the elevated-temperature sputter with a substrate temperature of 450 degrees C, and the substrate for polish was formed. The pressure was set to 4mTorr(s), using Ar as sputtering gas in aluminum-Cu membrane formation. Below, the elevated-temperature sputter aluminum-Cu film formed of the above-mentioned process is ground using the polish liquid of this invention, and the example which embedded aluminum-Cu in the wiring gutter and which embedded and formed wiring is explained.

[0034] The polish liquid used by introduction and this example is explained. The preparation odor of polish liquid prepared the neutral silica polish liquid which made pure water distribute the particle-size globular form silica particle of 10-40nm by the wet method which hydrolyzed tetraethyl orthochromatic silicate (TEOS) as an abrasive material particle in pure water first. Concentration of the silica particle used as an abrasive material particle was made into 10wt(s)%. The viscosity of this neutral silica polish liquid was 1.07cP(s) ( $1.07 \times 10^{-3}$ Pa-s). The polish liquid used actually was prepared by adding an electrolyte salt or an organic macromolecule in this neutral silica polish liquid.

[0035] As 1st polish liquid, the aluminium nitrate was added in the above-mentioned neutral silica polish liquid, and electrolyte addition neutral silica polish liquid was prepared. Aluminium nitrate concentration is made into 10-3 - 1wt% of within the limits, and is 0.5wt(s)% typically. Polish liquid viscosity increased to 5.09cP(s) ( $5.09 \times 10^{-3}$ Pa-s) by addition of an aluminium nitrate. That is, by adding an aluminium nitrate in neutral silica polish liquid, the silica particle was made to condense beforehand and polish liquid viscosity was enlarged about 4.5 times.

[0036] Moreover, as 2nd polish liquid, the cellulose was added in the above-mentioned neutral silica polish liquid, and macromolecule addition neutral silica polish liquid was prepared. Cellulose concentration is made into 10-6 - 10-1wt% of within the limits, and is  $4.7 \times 10^{-3}$ wt% typically. Polish liquid viscosity increased to 5.4cP(s) ( $5.40 \times 10^{-3}$ Pa-s) by addition of this cellulose. That is, by adding a cellulose in neutral silica polish liquid, the silica particle was made to condense beforehand and polish liquid viscosity was enlarged about 5 times.

[0037] The 1st of a more than and the 2nd polish liquid were used, and the substrate for polish which has the aluminum-Cu film on a front face was ground. The used polish equipment is the same as before,



and uses foaming polyurethane (Rodel Nitta CO.: IC-1000 laminating pad) as a scouring pad. Moreover, the speed of supply of 35rpm and polish liquid considered rotational speed of a polish head and a turn table as a part for 60ml/.

[0038] It is drawing showing the effect of the polishing pressure force exerted on a polish rate at drawing 1. Although the polish rate increased with the increment in the polishing pressure force, the dependency of an additive kind was not seen mostly. moreover, drawing 2 -- the polishing pressure force -- 0.4kg/cm<sup>2</sup> \*\*\*\*\* -- it is drawing showing the dependency of the polish rate to the cellulose concentration at the time of grinding using the 2nd polish liquid. In experiment within the limits whose cellulose concentration is 10<sup>-6</sup> - 10<sup>-1</sup>wt%, the polish rate is fixed at 0.2 micrometer/min, and the dependency of the polish rate to cellulose concentration is not seen. As for this, the same was said of the case of the 1st polish liquid, and the aluminium nitrate addition dependency was not accepted in a polish rate at experiment within the limits whose aluminium nitrate concentration is 10<sup>-3</sup> - 1wt%. As mentioned above, it was checked that the aluminum film can be ground by adding an electrolyte salt and an organic macromolecule in neutral silica polish liquid beforehand, and making it condense an abrasive material particle beforehand. In addition, although the 1st of this invention and the 2nd polish liquid were improving substantially as compared with conventional polish liquid, the stability of the polish property over progress of polish, for example, the stability of a polish rate, excelled [ direction / of the 1st polish liquid which added the electrolyte salt and dissolved the metal ion into polish liquid beforehand ] in stability.

[0039] Moreover, apart from polish of the above-mentioned aluminum film, the experiment which grinds silicon oxide using the 1st of this invention and the 2nd polish liquid was conducted. Consequently, the polish rate of the silicon oxide at the time of using the 1st polish liquid by aluminium nitrate addition was about [ in the case of the aluminum film ] about 1/10, and was about about 1/20 with the 2nd polish liquid by cellulose addition. That is, in spite of having been neutral polish liquid by using the 1st [ by this invention ], and 2nd polish liquid, it has checked that the selectivity of the aluminum polish to a substrate interlayer insulation film (here silicon oxide) was secured. However, it was checked that extent of selectivity is also excellent in the direction of the 2nd polish liquid by cellulose addition.

[0040] Drawing 3 is drawing [ polish before (immediately after membrane formation) / reflection factor / at the time of performing aluminum-Cu alloy film polish using the 1st polish liquid (aluminium nitrate addition, concentration 0.5wt%) by this invention, and the 2nd polish liquid (cellulose addition, concentration 4.7x10<sup>-2</sup>wt%) ]. In addition, polish of a metal membrane performed measurement of the reflection factor of drawing 3 in the phase (at namely, the event of the interlayer insulation film not being exposed) which advanced to the middle. Compared with the aluminum-Cu film front face immediately after elevated-temperature spatter membrane formation, even when which polish liquid is used, a reflection factor is larger than drawing 3 about a maximum of 7%. This is because the surface smoothness of the aluminum-Cu film improved by polish. When aluminium nitrate addition was compared with cellulose addition, near 300nm, compared with amyl nitrate addition neutral polish liquid, 10% or more reflection factor had the large reflection factor on the front face of aluminum which used cellulose addition neutral silica polish liquid, and it turned out that it excels by the smooth nature on the front face of aluminum after polish. In addition, even when changing cellulose concentration, in experiment [ 10<sup>-6</sup> - 10<sup>-1</sup>wt% of ] within the limits, it was changeless to the reflection factor of a polished surface.

[0041] Thus, since oxidizers, such as hydrogen peroxide solution added by conventional polish liquid, are not added in amyl nitrate addition neutral silica polish liquid and cellulose addition neutral silica polish liquid, the metal oxide-film metallurgy group hydroxylation film is not formed during polish in a surface of metal. Moreover, since polish liquid is neutrality, a metal membrane does not dissolve in polish liquid. Furthermore, blemish generating on the front face of a metal membrane is controlled by using the very small globular form abrasive material particle.

[0042] In addition, although it was the aluminum-Cu alloy with which the metal for polish of this example contained not pure aluminium but 0.5% of copper in relation to the 1st invention, only the

aluminium nitrate was used as an electrolyte salt added in the 1st polish liquid. The reason the stability of polish property sufficient also by this was acquired is because the content of the copper in an alloy is dramatically small. In general, when the content of addition metals other than the principal component in an alloy exceeds 5%, it is desirable to also double the electrolyte salt containing the addition metal, to add, and to prepare polish liquid. Of course, even when the content of the addition component metal in an alloy is 5% or less of minute amount, it cannot be overemphasized that the electrolyte salt may be added and polish liquid may be prepared.

[0043] Moreover, in embedding wiring structure which was made applicable to polish by this example, inserting barrier metal layers (for example, several nm in thickness to about several 10nm titanium etc.) between an interlayer insulation film and metal wiring is often performed. However, the thickness of such a barrier metal layer is usually dramatically thin, and since a barrier metal layer will be ground in a polish culmination just before an interlayer insulation film is exposed, fluctuation of the polish property by polish of a barrier metal layer does not usually pose a problem.

[0044]

[Example 2] In the above-mentioned example 1, even if it used any of the electrolyte salt addition polish liquid of this invention, and organic macromolecule addition polish liquid, polish of the stable metal membrane and selection polish of the metal membrane to an interlayer insulation film are possible, and it became clear that blemish generating in the front face after polish can be controlled. However, the difference in a polish property also existed between both polishes liquid, electrolyte addition polish liquid won in the stability of the polish property over progress of polish, and it became clear by one side that organic macromolecule addition polish liquid was also excellent by the selectivity over control and the interlayer insulation film of blemish generating on the front face of a metal membrane. If the above is taken into consideration, electrolyte addition polish liquid fits the activity at the flattening polish process from the early stages of polish. It is the process which this process carries out flattening of the irregularity on the front face of a metal membrane, and removes many parts of the metal membrane on an interlayer insulation film, and the stability of a polish property is searched for most. Moreover, the organic macromolecule addition abrasive material is suitable for the activity at the mirror-polishing process which removes a subsequent residual metal and exposes an interlayer insulation film. At this process, control of the high selection ratio in polish and blemish generating to a surface of metal is called for strongly.

[0045] So, in the example 2, flattening polish which used electrolyte addition polish liquid as the 1st step is performed, mirror polishing using organic macromolecule addition polish liquid as the 2nd step is performed after that, it continues further, a washing process is performed as the 3rd step, and the example which embedded according to a series of above processes, and formed metal wiring is explained.

[0046] The outline of the polish equipment used by this example is shown in drawing 4. The polish process in this example has three steps as above-mentioned. The polish equipment of this example has three polish stages corresponding to each of this step, and while it exposes an interlayer insulation film for the flattening polish process of removing the outline of a metal membrane on the 1st polish stage 10 on the 2nd polish stage 20, it can perform a substrate washing process for the high selection mirror-polishing process which finishes a metal membrane front face on the 3rd polish stage 30. Each polish stage consists of the same configuration fundamentally, and it has the turn tables 11, 21, and 31 which equipped the top face with the scouring pad, respectively, the polish heads 12, 22, and 32, the load impression devices 13, 23, and 33, and the polish liquid feeder styles 14, 24, and 34. It can be made to be able to rotate at the rate of arbitration by the rolling mechanism, respectively, and a turn table and a polish head can apply the load of arbitration to a polish head according to a load impression device in that case. The substrates 15, 25, and 35 used as the object for polish are held by making a front face into facing down at a polish head, and perform polish or washing of a substrate by supplying polish liquid 16, 26, and 36 from a polish liquid feeder style. In addition, although the key objective of the process in the 3rd polish stage is washing of a substrate, the expression with polish liquid is used here.

[0047] In the polish equipment used by this example, the above the 1st - 3rd polish stage are arranged on

one susceptor 1, and really have composition. Moreover, the substrate before polish is first installed in a loader 2, it is conveyed one by one according to the automatic conveyance device which is not illustrated after that on the 1st - the 3rd polish stage, the polish process of a three-stage is performed, and the substrate after polish / washing termination is stored in an unloader 3. Hereafter, the substrate in the polish location on each stage is distinguished as substrates 15, 25, and 35, respectively.

[0048] The substrate made applicable to polish is almost the same as the substrate used in the example 1. That is, the wiring gutter with a depth of 500nm is beforehand formed in the interlayer insulation film, and after that, by the elevated-temperature spatter with a substrate temperature of 450 degrees C, aluminum-Cu0.5% (700nm in thickness) is embedded, and membranes are formed. The membrane formation conditions of an aluminum-Cu alloy are the same as an example 1. There is surface irregularity reflecting the level difference of a substrate wiring gutter of about 300nm in this aluminum-Cu alloy film upper part.

[0049] Hereafter, the polish process in the 1st, 2nd, and 3rd polish stage in drawing 4 is explained.

[0050] The polish process in the 1st polish stage is a flattening polish process of removing the surface irregularity of an aluminum-Cu alloy film front face. the polish liquid 16 used here distributes the particle-size globular form silica particle of 40-80nm depending on the method of wet to pure water as an abrasive material particle -- making -- further -- an aluminium nitrate -- 0.5wt(s)% -- it is the added electrolyte addition polish liquid. Concentration of a silica particle was made into 10 - 20wt%. a scouring pad -- a with a degree of hardness of about (JIS-A specification and the following -- the same) 90 to 100 hard scouring pad -- using -- the load impression device 13 -- 0.3-0.4kg/cm<sup>2</sup> The high polishing pressure was impressed. The rotational frequency of a turn table 11 and the polish head 12 was set to 30 - 90rpm. This process is a process which carries out polish clearance of a lot of aluminum-Cu alloy film, and the rapidity of polish is dramatically important for it. For this reason, large polish particle and hard pad of particle size are used above a little, and it is grinding under the conditions of high voltage impression and a high-speed revolution. By the above configuration, it ground until the aluminum residual membrane was set to 100nm.

[0051] The polish process in the 2nd polish stage is a high selection mirror-polishing process which controls polish of a substrate interlayer insulation film as much as possible, and carries out mirror polishing of the aluminum film selectively. as the polish liquid 26 of this process -- a 10nm - 40nm globular form silica particle -- 10 - 20wt% -- the distributed pure water -- a cellulose -- 0.1wt(s)% -- using the added organic macromolecule addition polish liquid, mirror polishing of the aluminum-Cu alloy was carried out until the substrate interlayer insulation film appeared with the scouring pad of an elasticity pad (degrees of hardness 60-90). The pressure impressed according to the load impression device 23 at this time is 0.3kg/cm<sup>2</sup>. It considered as the following low voltage force, and the rotational frequency of a turn table 21 and the polish head 22 was set to 10 - 40rpm, respectively. Here, a particle and an elasticity pad with a small particle size are used, and in order to raise the smooth nature of the polished surface in the aluminum-Cu alloy film after polish, it grinds for making selectivity of polish higher under the conditions of low voltage and a low-speed revolution. According to this process, embedding aluminum wiring (pellet SHIN aluminum wiring) without surface irregularity is obtained.

[0052] The polish process in the 3rd stage is a process which washes the compound front face which was formed of the polish process to the 2nd stage, and which consists of aluminum-Cu film and an interlayer insulation film. Pure water was used as polish liquid 36 used at this process. Moreover, the impressed pressure as a scouring pad, use a with a degree of hardness of 60 or less elasticity pad, and according to the load impression device 33 is 0.3kg/cm<sup>2</sup>. It ground hereafter on the conditions of 40 or less rpm of rotational frequencies of a turn table 31 and a polish head. In addition, as polish liquid 36 used at this process, what added organic macromolecules (surfactant), such as a cellulose not more than 0.1wt%, may be used for pure water.

[0053] At a series of above polish processes, DAMASHIN wiring with which the aluminum-Cu alloy with a thickness of 500nm was embedded at the interlayer insulation film was formed. The irregularity of this aluminum-Cu wiring front face is 10nm or less, and excelling in smooth nature was checked.

[0054] In this example, as shown in drawing 4, the 1st to the 3rd made polish stage connection, and the

polish equipment which can carry out automatic conveyance of the substrate between stages was used. However, even if it prepares two or more polish equipments according to individual and makes it make the 1st, 2nd, and 3rd polish process perform to each, the effectiveness of this invention is completely the same. Moreover, important processes are the 1st and 2nd polish processes, and are intrinsically [ among the above three processes ] good also by another washing means. [ of the 3rd washing process ]

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[Translation done.]

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3. In the drawings, any words are not translated.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is drawing showing the polish rate of the aluminum-Cu alloy film and the relation of the polishing pressure force with the polish liquid of this invention.

[Drawing 2] It is drawing showing the relation between the organic macromolecule concentration in the 2nd [ of this invention ] polish liquid, and the polish rate of the aluminum-Cu alloy film.

[Drawing 3] It is drawing showing the reflection factor of the aluminum-Cu alloy ground with polish liquid to this invention.

[Drawing 4] It is the process sectional view showing the embedding wiring formation polish approach by this invention.

[Drawing 5] It is drawing showing conventional polish equipment.

[Drawing 6] It is the process sectional view showing the conventional embedding wiring formation process.

[Drawing 7] It is drawing showing the time dependency of the polish head torque in the conventional polish approach.

[Description of Notations]

- 1 Susceptor
- 2 Loader
- 3 Unloader
- 10 1st Polish Stage
- 20 2nd Polish Stage
- 30 3rd Polish Stage
- 11, 21, 31 Turn table
- 12, 22, 32 Polish head
- 13, 23, 33 Load impression device
- 14, 24, 34 Polish liquid feeder style
- 15, 25, 35 Substrate
- 16, 26, 36 Polish liquid
- 61 Substrate
- 62 Lower Layer Wiring
- 63 Interlayer Insulation Film
- 64 Beer Hall
- 65 Metal Membrane

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[Translation done.]

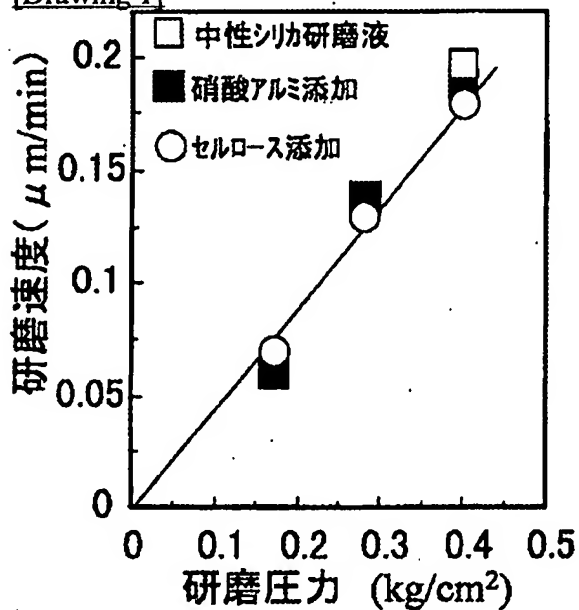
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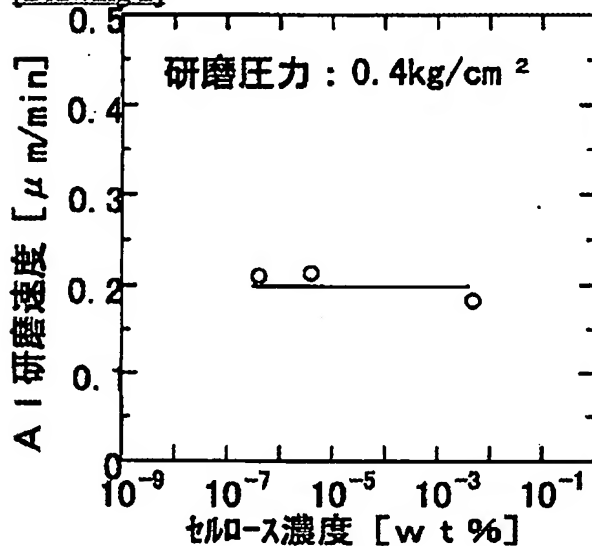
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## DRAWINGS

[Drawing 1]

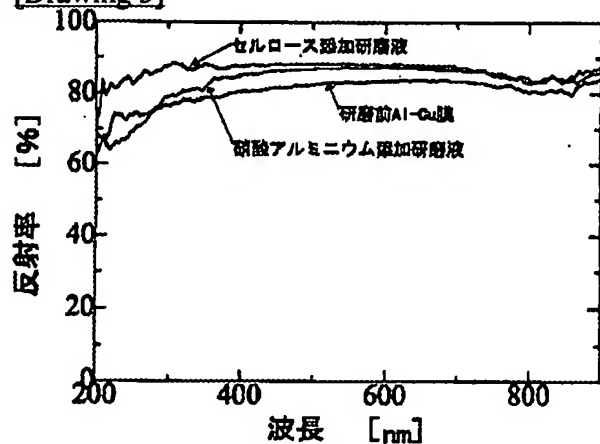


[Drawing 2]

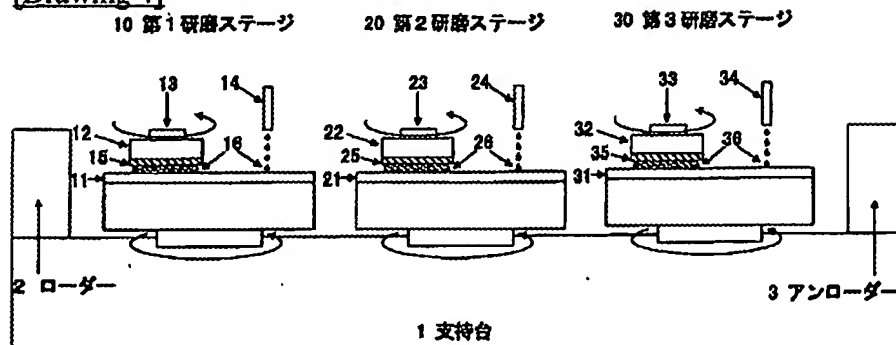




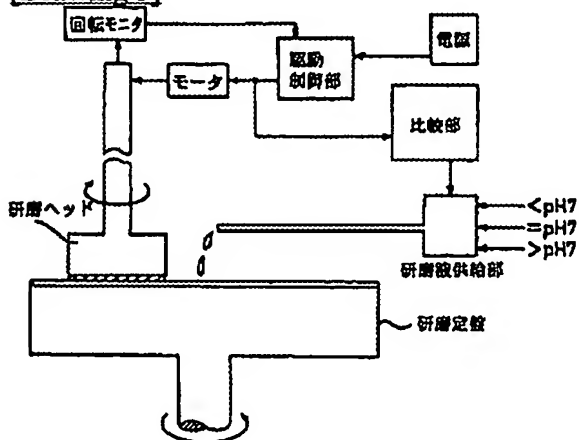
[Drawing 3]



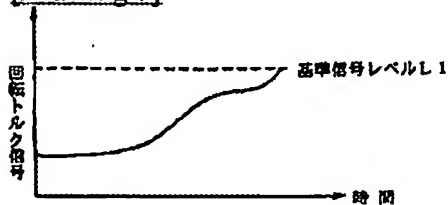
[Drawing 4]



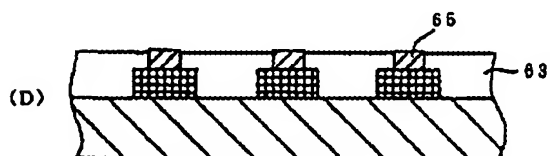
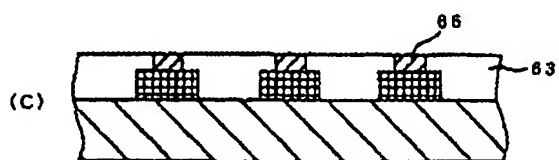
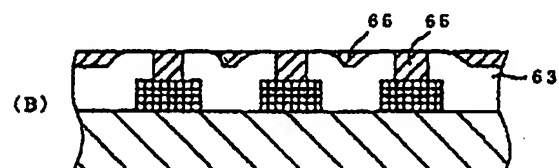
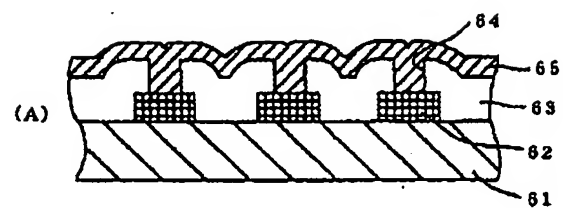
[Drawing 5]



[Drawing 7]



[Drawing 6]



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[Translation done.]

103-1<sup>st</sup>

Takumi

DERWENT- 1999-319132  
ACC-NO:

DERWENT- 200213  
WEEK:

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TITLE: Polishing liquid for insulating semiconductor substrate - has aprotic solvent containing lapping powder particles to which electrolytic salt solution is added

PRIORITY-DATA: 1997JP-0266643 (September 30, 1997)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
✓ JP <u>11111657</u>	A April 23, 1999	N/A	010	H01L 021/304
JP 3255095	B2 February 12, 2002	N/A	009	H01L 021/304

INT-CL (IPC): B24B037/00, C09K003/14 , H01L021/304

ABSTRACTED-PUB-NO: JP 11111657A

BASIC-ABSTRACT:

NOVELTY - The liquid includes an aprotic solvent containing lapping powder particle. An electrolytic salt solution containing principal component of the metal surface to be polished is added to the aprotic solvent.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for polishing method.

USE - For polishing semiconductor substrates on which wiring is performed.

ADVANTAGE - Smooth insulating surface is obtained. Superior metal sanding is obtained as the lapping powder is condensed beforehand on the metal surface.

DESCRIPTION OF DRAWING - The figure shows the implanting wiring formation polishing method.

(19)



JAPANESE PATENT OFFICE

## PATENT ABSTRACTS OF JAPAN

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(71) Applicant: NEC CORP

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(72) Inventor: NAKAJIMA TSUTOMU

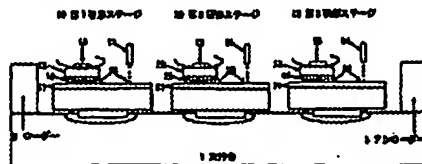
(54) POLISHING LIQUID AND POLISHING METHOD

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(57) Abstract:

**PROBLEM TO BE SOLVED:** To improve the stability of polishing characteristics and selectivity to a substratum insulating film by dispersing abrasive agent particles in neutral polishing liquid in water, and adding electrolyte salt containing metal element constituting a metal film to be polished.

**SOLUTION:** In a polishing process on a first polishing stage 10, spherical silica particles are dispersed in pure water by using polishing liquid 16 as abrasive agent particles, and surface unevenness of an Al-Cu alloy film surface is eliminated by using electrolyte-added polishing liquid in which nitrate aluminum of 0.5 wt.% is added. In a polishing process on a second polishing stage 20, polishing of an intersubstratum layer insulating film is restrained by using abrasive liquid 26, and an aluminum film is selectively subjected to mirror surface polishing. A composite surface constituted of an Al-Cu film and an inter-layer insulating film which are formed in polishing processes as far as a third stage 20 is cleaned.



(10-40) nm  
granular  
silica

(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平11-111657

(43) 公開日 平成11年(1999) 4月23日

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B 2 4 B 37/00		B 2 4 B 37/00 Z
C 0 9 K 3/14	5 5 0	C 0 9 K 3/14 5 5 0 Z

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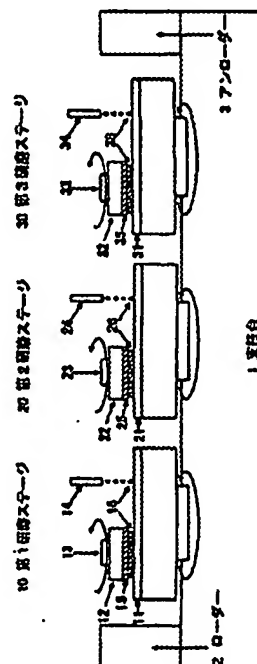
(74) 代理人 弁理士 京本 直樹 (外2名)

(54) 【発明の名称】 研磨液および研磨方法

(57) 【要約】

【課題】 絶縁膜上の配線溝に埋め込んだ金属膜を研磨して埋め込み配線を形成する方法において、金属研磨面の変質や傷発生、研磨特性の変動を防止する。

【解決手段】 研磨対象金属を含有する電解質塩を添加した中性研磨液を使用して金属膜を平坦化研磨する。この研磨液により、研磨特性が安定で傷も少ない研磨が可能である。引き続き、セルロース等の有機高分子を添加した中性研磨液を使用して研磨を行う。この研磨液により、高選択の鏡面研磨が可能である。以上の工程により、傷発生が少なく、かつ安定した埋め込み配線の形成が可能となる。



## 【特許請求の範囲】

【請求項1】金属研磨用の研磨液において、中性溶媒中に研磨剤粒子を分散させ、さらに少なくとも研磨対象金属における主成分元素を含む電解質塩を添加したことを特徴とする研磨液。

【請求項2】前記電解質塩が研磨対象金属における主成分元素の硝酸塩、硫酸鉛、塩化物、酢酸塩、またはリン酸塩であることを特徴とする請求項1に記載の研磨液。

【請求項3】前記中性溶媒が水であり、かつ前記研磨剤粒子が単分散の球形微粒子であることを特徴とする請求項1または請求項2に記載の研磨液。

【請求項4】研磨剤粒子を水に分散させた研磨液に水溶性有機高分子を添加したことを特徴とする研磨液。

【請求項5】前記水溶性有機高分子の濃度が1wt%以下であることを特徴とする請求項4に記載の研磨液。

【請求項6】前記水溶性有機高分子がセルロースであることを特徴とする請求項4または請求項5に記載の研磨液。

【請求項7】前記研磨剤粒子が単分散の球形微粒子であることを特徴とする請求項4から請求項6のいずれかに記載の研磨液。

【請求項8】半導体基板上に絶縁膜を介して形成された金属膜の研磨方法において、請求項1から請求項7のいずれかに記載の研磨液を使用することを特徴とする研磨方法。

【請求項9】あらかじめ配線溝が形成された絶縁膜上の該配線溝に埋め込んで成膜した金属膜の研磨方法において、請求項1から請求項7のいずれかに記載の研磨液を使用して前記絶縁膜より上部にある金属膜を研磨除去し、前記絶縁膜と前記配線溝に埋め込まれた金属膜よりなる複合表面を露出することを特徴とする研磨方法。

【請求項10】あらかじめ配線溝が形成された絶縁膜上の該配線溝に埋め込んで成膜した金属膜の研磨方法において、請求項1から請求項3のいずれかに記載の研磨液を使用して前記絶縁膜より上部にある金属膜の概略を研磨する第1の研磨工程と、引き続き請求項4から請求項7のいずれかに記載の研磨液を使用して前記絶縁膜より上部にある金属膜を研磨して前記絶縁膜と前記配線溝に埋め込まれた金属膜よりなる複合表面を露出する第2の研磨工程とを有することを特徴とする研磨方法。

【請求項11】前記第2の研磨工程に引き続き、前記複合表面を洗浄する第3の工程をさらに有することを特徴とする請求項10に記載の研磨方法。

## 【発明の詳細な説明】

## 【0001】

【発明が属する技術分野】本発明は、半導体装置の配線形成方法のうち特に配線材料の研磨方法およびそこに使用する研磨液に関するものであり、詳しくは層間絶縁膜に形成された配線溝に金属を埋め込むことで配線を形成する際の研磨方法および研磨液に関するものである。

## 【0002】

【従来の技術】トランジスタの微細化に伴って、アルミ等の金属配線の微細化が必要とされているが、従来の金属膜のドライエッチングを用いた配線形成では、エッチングガスによる金属コロージョン（金属腐食）による配線信頼性劣化が問題となっている。そこで、あらかじめ層間絶縁膜に配線溝や下地配線へのビアホールを形成しておき、高温スパッタ法やCVD法でかかる配線溝やビアホールを埋め込みながら層間絶縁膜全面に金属膜を成長し、この層間絶縁膜上の金属膜を化学機械研磨（CMP）等の選択研磨によって除去することにより、配線溝やビアホールへの埋め込み金属配線を形成する方法が注目されている。上記工程により形成される金属埋め込み型の配線は、ダマシン配線と呼ばれている。

【0003】ダマシン配線には、ドライエッチングによる加工が困難な材料の適用や、配線溝に対して自己整合的に形成されたビアホールに配線金属を埋め込んだ配線／ビアの一体化構造が可能になるという利点がある。一方、アルミニウムのような軟質金属を研磨する際には、研磨後の金属表面の加工凹凸が懸念される。このような研磨後の金属表面の性状は、研磨圧力、研磨パッド回転数、研磨パッド硬度や研磨に用いる研磨液（スラリー）の特性が複雑に絡み合った研磨因子に影響を受ける。

【0004】図5に、従来のCMP装置の一例を示す。従来のCMP装置は、基板を吸着する研磨ヘッド、研磨定盤、研磨ヘッドの回転トルク計測部および研磨液供給部からなる。研磨ヘッドの駆動は、前記回転トルク計測部からのトルク信号により制御され、研磨液供給部はアルカリ性（ $\text{pH} > 7$ ）、中性（ $\text{pH} = 7$ ）および酸性（ $\text{pH} < 7$ ）の各研磨液を供給可能である。

【0005】図6に、CMP装置を用いたダマシン配線形成プロセスの一例を示す。以下、図6を参照して、該プロセスを工程順に簡単に説明する。

【0006】あらかじめ基板61上に下層配線62及び層間絶縁膜63を形成し、さらにビアプラグを埋め込むためのビアホール64を開口しておく。その後、層間絶縁膜63上の全面に、金属膜65（例えばアルミニウム）を成膜する（図6（A））。この金属膜65の成膜手法には化学気相成長（CVD）法やスパッタ法などを用いることが可能であるが、埋め込み性の良好な成膜手法によりビアホール64内に金属膜65を埋め込む必要がある。

【0007】次に、金属研磨用の研磨液を用いて金属膜65を研磨する（図6（B））。この金属研磨用の研磨液としては、例えば酸性（ $\text{pH} = 3$ 程度）のアルミナスラリー等が用いられる。このような酸性研磨スラリーは、一般に金属膜の研磨速度が層間絶縁膜に対して大きいという特徴を有する。本工程における金属膜研磨の終点検知は、図5の研磨装置において駆動制御部からの回転トルク出力信号をモニタすることにより行う。これ



は、酸性研磨スラリーによる研磨において回転数を一定として研磨を行う場合、研磨面に層間絶縁膜63が現れると研磨ヘッドの回転トルクが急上昇することを利用している。すなわち、図7に示すように、研磨の進行に伴い回転トルクが上昇し、あらかじめ設定したトルク信号出力のレベルを越えた時点で金属研磨工程を終了する。この時点では、図6(B)に示す通り、層間絶縁膜13上には金属膜65が一部残存している。

【0008】引き続き、シリカ粒子を分散させた中性シリカスラリーを用いて、金属膜65と層間絶縁膜63とをさらに研磨する(図6(C))。この中性シリカスラリーは、金属膜と同時に層間絶縁膜をも研磨するという特徴を有する。その結果、層間絶縁膜上に残存する金属膜65と層間絶縁膜63の平坦化が同時に進行する。

【0009】最後に、研磨液を中性シリカスラリーから水酸化カリウム等を添加したアルカリ性シリカスラリーに切り替えて更に研磨を続ける。このアルカリ性シリカ研磨液は、層間絶縁膜の研磨速度が金属膜に対して大きい特徴を有することから、研磨を続けることで、金属膜65のプラグを層間絶縁膜63から突出させることができる。この後に、上層配線金属の成膜・パターニングを行うことにより、多層配線構造が形成される。

【0010】以上述べた従来技術は、例えば特開平8-124886号公報に記載されている。

【0011】

【発明が解決しようとする課題】以上述べた従来の研磨方法においては、以下に述べる課題があった。

【0012】まず、従来の研磨液を用いた金属研磨においては、研磨後の金属表面に変質層が残留するという問題があった。従来の金属研磨用研磨液には、例えばアルミナ粒子を分散させた酸性スラリーを用いるが、このスラリーには通常過酸化水素水等の酸化剤が含まれており、金属表面に金属水酸化物あるいは酸化物を形成する。すなわち、金属研磨の進行は、研磨スラリー中の酸化剤と金属との反応で金属表面に金属水酸化物(あるいは酸化物)変質層を形成し、この表面の変質層を研磨パッドと研磨粒子(ここでは、アルミナ粒子)とで削り取ってゆくことで行われるのである。このため必然的に、研磨後の金属表面も金属水酸化物または酸化物で覆われることになる。このような金属表面の変質層は多くの場合絶縁性であるため、研磨後の金属上に上層金属配線を形成する際に、接点抵抗が増大をもたらすという問題があった。また、この金属表面に形成された水酸化物(または酸化物)は、場合によってはその一部が剥がれ落ち、研磨後の表面が平滑でなくなるといった問題を引き起こす。このような剥がれにより金属配線表面に局所的な凹部が存在すると、その領域から断線する場合もあり、配線信頼性の著しい低下を引き起こす。

【0013】また、従来の研磨液を用いた金属研磨においては、その研磨速度等の研磨特性が、研磨の進行と

もに変動してしまうという問題があった。金属膜の研磨特性は、研磨液中の研磨粒子の凝集状態に大きく依存する。この研磨剤粒子の凝集状態は研磨液中の電解質濃度やpHにより変化するため、金属膜研磨の進行とともに研磨液中の成分と金属が反応する、あるいは研磨液中に金属が溶解する等により研磨液の物性が変化すると、研磨の初期と終期とで研磨特性が変化してしまうのである。従来の研磨液では、酸化剤を含む酸性の金属研磨用スラリーを使用していたため、研磨液中への金属膜あるいはその水酸化物等の溶解が顕著であり、スラリー中の金属イオン濃度は研磨進行に伴って増大する傾向を持っていた。一般に、スラリー中の電解質濃度が増大すると研磨剤粒子の周りに存在する電気二重層の厚みが減少して、研磨剤粒子の凝集が生じ易くなる。その結果、研磨スラリーの粘度が研磨の進行に伴って増大してしまうといった課題があった。

【0014】以上の問題は、金属研磨用の研磨スラリーを単に中性にすることだけでは回避することが出来ない。これは、研磨スラリーを中性にすることによって研磨中の金属膜の研磨スラリーに対する溶解を抑制することは可能であるが、その一方で下地層間絶縁膜の研磨速度が上昇し、絶縁膜に対する金属膜の選択研磨性が十分にとれなくなるといった問題が有るためである。この場合、研磨工程の管理は非常に困難となる。

【0015】本発明は、以上の課題を解決するためになされたものであり、金属の研磨における研磨特性の安定性、下地絶縁膜に対する選択性を向上し、また同時に金属表面への傷発生を防止することを目的とし、ひいては研磨工程を用いて製造される半導体装置の信頼性を向上することを目的としている。

【0016】

【課題を解決するための手段】かかる技術課題を解決する手段として、第1の発明では、金属膜を研磨する方法において、研磨剤粒子を水に分散させ、さらに研磨対象となる金属膜を構成する金属元素を含む電解質塩を添加した中性研磨液、およびこの研磨液を用いた研磨方法を提供する。

【0017】また、第2の発明では、研磨剤粒子を水に分散させ、さらに水溶性有機高分子を添加した中性研磨液、およびこの研磨液を用いた研磨方法を提供する。研磨液中の水溶性有機高分子としては、例えばセルロースが使用可能であり、その濃度は1wt%以下であることが望ましい。

【0018】この第1および第2の発明において使用する研磨剤粒子は、単分散の球形微粒子であることが望ましい。

【0019】また、本発明は、配線溝を有する絶縁膜上に埋め込み成膜された金属膜を研磨する方法において、まず第1の発明による研磨液を用いて金属膜の概略を研磨除去し、さらに引き続き第2の発明による研磨液を用

いて絶縁膜の露出および金属膜の鏡面研磨を行う研磨方法を開示する。さらにその後には、露出した絶縁膜と配線溝に埋め込まれた金属膜からなる複合表面を洗浄する工程を有してもよい。

【0020】(作用) 本発明においては、中性の研磨液に研磨対象となる金属を含む電解質塩、もしくは有機高分子を溶解させることにより研磨を行う。その基本となる中性研磨液とは、典型的には純水に研磨粒子(シリカ等)を分散させただけのものであるため、研磨する金属表面には酸化物や水酸化物等の皮質層は形成されない。この場合、金属と研磨粒子の直接の接触により研磨が進行する。なお、厳密には中性とは $pH=7$ のことを意味するが、本発明でいう中性研磨液とは $pH6\sim8$ 程度の研磨液であり、従来の $pH=3$ 程度の酸性研磨液や $pH=9\sim11$ 程度のアルカリ性研磨液と区別するためのものである。

【0021】第1の発明においては、半導体基板上の金属膜を研磨する際、酸化剤を添加しない研磨液を用いることにより、金属表面に絶縁体である金属水酸化膜や金属酸化物を形成することなく研磨することが可能となる。また、研磨液にあらかじめ研磨対象となる金属の硝酸塩、硫酸塩や塩化物等の電解質塩を溶解しておくことで、金属研磨進行にともなう研磨液中の金属イオン濃度の変化を抑制することが可能となる。これにより、研磨液中の研磨剤粒子の凝集状態が変化することが少なくなり、金属研磨の速度安定性が増加するという作用を有する。

【0022】また、第2の発明においては、酸化剤を添加しない研磨液に水溶性有機高分子を溶解させてあらかじめ研磨剤粒子を凝集させておくことにより、金属研磨の速度安定性を増すことが可能となる。さらに、水溶性有機高分子は金属膜下の層間絶縁膜に選択吸着して有機膜の表面保護膜を形成するため、層間絶縁膜に対する金属膜の研磨選択性を向上させるという効果も有する。この水溶性有機高分子の添加量は、層間絶縁膜表面に極薄い有機膜を形成するだけでよいので、 $1wt\%$ 以下程度の微量添加においても十分にその機能を有する。

【0023】これら第1、第2の発明による研磨液を用いた場合、金属研磨面には金属酸化膜や金属水酸化膜が形成されることなく、研磨剤粒子と金属との直接接触により研磨が進行する。このような場合に金属表面の傷発生を防止するには、研磨粒子として粒子同士の固結のない単分散の球形粒子を使用することが極めて効果的である。

【0024】また、層間絶縁膜に形成された配線溝上の金属膜を研磨して金属膜を埋め込むダマシン配線の形成方法においては、第1の研磨工程として研磨安定性の高い金属膜の電解質塩を添加した研磨液を用いて金属膜の主なる部分を平坦化研磨し、引き続き第2の研磨工程として層間絶縁膜との選択比の高い水溶性有機高分子を添

加した研磨液を用いて層間絶縁膜上に残存する金属膜を除去する研磨方法が有効である。さらに第3工程として研磨剤を含まない中性洗浄液を滴下して洗浄基板表面の洗浄を行ってもよい。

【0025】

【発明の実施の形態】第1の発明の実施の形態においては、金属研磨に用いる研磨液中にあらかじめ研磨対象となる金属膜と同一金属の硝酸塩、硫酸塩等の電解質塩を予め研磨液に添加した研磨液を使用して研磨を行う。また、従来金属研磨においては、過酸化水素水等の酸化剤を添加して、金属表面を酸化あるいは水酸化してこの表面反応層を研磨により除去することが通常であったが、本発明の研磨液においては研磨液に酸化剤を添加せず、中性研磨液中の研磨剤で直接金属膜を研磨する。研磨に使用する装置構成は、従来通りのものでよい。

【0026】研磨液中への上記電解質塩の溶解により、研磨剤粒子を予めある程度凝集させておくことが可能となる。これにより研磨中の金属膜の研磨液への溶解を抑制し、金属イオン濃度上昇による研磨液の粘度の上昇と、それに伴う研磨速度等の研磨特性の変動を回避する。また、同時に研磨粒子の凝集により、層間絶縁膜に対する金属膜研磨の選択性が得られる。上記電解質塩としては、例えば研磨対象となる金属膜がアルミニウムの場合、硝酸アルミニウム、硫酸アルミニウム、塩化アルミニウム、酢酸アルミニウムやリン酸アルミニウムなどを使用する。また金属膜が銅の場合、硝酸銅、硫酸銅、塩化銅、酢酸銅やリン酸銅などである。つまり、研磨液に研磨対象となる金属膜のイオンをあらかじめ溶解していることが肝要である。

【0027】研磨対象となる金属膜が合金である場合は、その合金を構成する元素全ての金属イオンを研磨液中に溶解しておくことが最も望ましい。例えば、 $Al-Cu$ 合金の研磨を行う場合は、中性研磨液中に例えば硝酸アルミニウムと硝酸銅の両者をあらかじめ溶解させておく。ただし、研磨対象金属が、主成分となる金属に微量の他種金属を添加したような合金の場合は、主成分金属のイオンを中性研磨液中に溶解させるだけでもよい。おおむね、主成分金属の組成比が $95\%$ 以上の場合には、研磨液中には主成分金属イオンを溶解させるだけでよい。例えば、半導体装置の配線に多用される銅の組成比が $1\%$ 程度以下の $Al-Cu$ 合金の研磨を行う場合には、研磨液中にはアルミニウムを含有する電解質塩のみを溶解しておけばよいのである。

【0028】研磨剤粒子としては、例えばテトラエチルシリケート(TEOS)の加水分解による液層析法(湿式法)で得た粒径 $10\sim300nm$ の微細球形シリカ粒子等の、粒子同士の焼結のない単分散の球形粒子を使用することが望ましい。本発明においては、研磨液に酸化剤を添加しないため、金属表面に水酸化物等の皮質層が形成されることがなく、研磨剤粒子と金属との直接

接触が生じる。この際、研磨剤粒子としては単分散の球形研磨剤粒子を用いることにより、金属膜表面への傷の発生を大幅に低減できるのである。上記微細球形シリカ粒子以外では、アルミニウム、チタン、ジルコニウムのアルコキシドを加水分解して得られるアルミナ粒子、酸化チタン粒子、酸化ジルコニア等の酸化物粒子（それぞれ粒子径：10nm～100nm程度）を用いても同様の効果が得られる。なお、研磨剤として塩化シリコンの気相熱分解によるシリカ粒子（一般には、ヒュームドシリカと呼ばれる）を用いることも可能ではあるが、特にアルミや銅といった柔らかい金属膜を研磨する場合の傷発生防止の観点からは、上述の湿式法による単分散球形粒子を用いて研磨することが望ましい。

【0029】第2の発明の実施の形態においては、研磨剤粒子を予めある程度凝集させておく別の手法として、水溶性の有機高分子、例えばセルロースを研磨剤に添加しておく。セルロース等の有機高分子は研磨剤粒子であるシリカ粒子の表面に吸着し、セルロース分子の絡み合いでシリカ粒子の連鎖を生じさせる作用があり、その結果として研磨液粘度が増加する。この場合、シリカ粒子が直接凝集するのでなく、セルロース等の高分子鎖の絡み合いで間接的に凝集した状態となる。

【0030】また、セルロース等の有機高分子は、研磨対象基板上でシリコン酸化膜やシリコン窒化膜、シリコン酸窒化膜等の層間絶縁膜に選択的に吸着し、その一方金属膜表面には吸着しない。このため、層間絶縁膜表面が有機高分子膜で覆われて、層間絶縁膜の研磨を抑制しつつ、金属膜の研磨を進めることが出来る。従って、この研磨液を使用する研磨では、金属膜表面に傷等が発生せざることなく直接金属膜を研磨し、かつ下地層間絶縁膜に対する高選択比を持たせることが可能となる。この水溶性有機高分子を添加した研磨液は、下地層間絶縁膜が現れる研磨最終段階で用いると効果的である。また、このような効果はアルミニウム、銅、タングステン、チタンといった金属膜のみならず、窒化チタンや窒化タングステンといった導電性窒化膜、タングステンシリサイドやチタンシリサイドといった金属シリサイド膜を研磨する際にも有効である。

【0031】上記水溶性有機高分子としては、セルロースの他にグリセリンやアクリル酸エステル等が使用可能である。また、この第2の発明の実施の形態において使用する研磨剤粒子は、第1の発明の場合と全く同様の理由により、湿式法による微細球形シリカ粒子等の単分散球形粒子を使用することが望ましい。

【0032】以下、図面を参照しつつ、本発明の実施例を詳細に説明する。

【0033】

【実施例1】あらかじめ層間絶縁膜に配線溝を形成しておき、その後基板温度450℃の高温スパッタ法でAl-CuO、5%（厚さ800nm）を埋め込み成膜し、

研磨対象基板を形成した。Al-Cu成膜におけるスパッタガスとしてはArを用い、圧力は4mTorrとした。以下では、上記工程により形成された高温スパッタAl-Cu膜を、本発明の研磨液を使用して研磨し、配線溝にAl-Cuを埋め込んだ埋め込み配線を形成した実施例を説明する。

【0034】初めに、本実施例で使用した研磨液について説明する。研磨液の調製においては、まず純水中に研磨剤粒子としてテトラエチルオルソシリケート（TEOS）を加水分解した湿式法による粒径10～40nm球形シリカ粒子を純水に分散させた中性シリカ研磨液を調製した。研磨剤粒子として用いるシリカ粒子の濃度は1.0wt%とした。この中性シリカ研磨液の粘度は1.07cP（ $1.07 \times 10^{-3} \text{Pa} \cdot \text{s}$ ）であった。本中性シリカ研磨液に電解質塩、あるいは有機高分子を添加することにより、実際に使用する研磨液を調製した。

【0035】第1の研磨液として、上記中性シリカ研磨液に硝酸アルミニウムを添加し、電解質添加中性シリカ研磨液を調製した。硝酸アルミニウム濃度は10<sup>-3</sup>～1wt%の範囲内とし、典型的には0.5wt%である。硝酸アルミニウムの添加で研磨液粘度は5.09cP（ $5.09 \times 10^{-3} \text{Pa} \cdot \text{s}$ ）に増加した。すなわち、中性シリカ研磨液に硝酸アルミニウムを添加することで、予めシリカ粒子を凝集させて研磨液粘度を4.5倍程度大きくさせておいた。

【0036】また、第2の研磨液として、上記中性シリカ研磨液にセルロースを添加し、高分子添加中性シリカ研磨液を調製した。セルロース濃度は10<sup>-6</sup>～10<sup>-1</sup>wt%の範囲内とし、典型的には4.7×10<sup>-3</sup>wt%である。このセルロースの添加で研磨液粘度は5.4cP（ $5.40 \times 10^{-3} \text{Pa} \cdot \text{s}$ ）に増加した。すなわち、中性シリカ研磨液にセルロースを添加することで、予めシリカ粒子を凝集させて研磨液粘度を5倍程度大きくさせておいた。

【0037】以上の第1及び第2の研磨液を使用して、Al-Cu膜を表面に有する研磨対象基板の研磨を行った。使用した研磨装置は従来通りのものであり、研磨パッドとしては発泡ポリウレタン（ロデールニッタ社：IC-1000積層パッド）を用いている。また、研磨ヘッドおよび研磨定盤の回転速度を35rpm、研磨液の供給速度は60ml/分とした。

【0038】図1に、研磨速度に及ぼす研磨圧力の影響を示す図である。研磨速度は研磨圧力の増加とともに増大するが、添加物種の依存性はほぼみられなかった。また、図2は、研磨圧力が0.4kg/cm<sup>2</sup>として第2の研磨液を用いて研磨した場合の、セルロース濃度に対する研磨速度の依存性を示す図である。セルロース濃度が10<sup>-6</sup>～10<sup>-1</sup>wt%の実験範囲内では、研磨速度は0.2μm/minで一定であり、セルロース濃度に対する研磨速度の依存性はみられない。これは第1の研

磨液の場合も同様で、硝酸アルミニウム濃度が $10^{-3} \sim 1 \text{ wt} \%$ の実験範囲内においては、研磨速度に硝酸アルミニウム添加量依存性は認められなかった。以上より、中性シリカ研磨液に予め電解質塩や有機高分子を添加して研磨剤粒子を予め凝集させておくことで、アルミニウム膜を研磨できることが確認された。なお、研磨の進行に対する研磨特性の安定性、例えば研磨速度の安定度は、従来の研磨液と比較して本発明の第1、第2の研磨液ともに大幅に向上していたが、電解質塩を添加してあらかじめ金属イオンを研磨液中に溶解した第1の研磨液の方が安定性に優れていた。

【0039】また、上記アルミニウム膜の研磨とは別に、本発明の第1、第2の研磨液を用いてシリコン酸化膜を研磨する実験を行った。その結果、硝酸アルミニウム添加による第1の研磨液を用いた場合のシリコン酸化膜の研磨速度はアルミニウム膜の場合の約10分の1程度であり、またセルロース添加による第2の研磨液では約20分の1程度であった。つまり、本発明による第1、第2の研磨液を用いることにより、中性の研磨液であるにも関わらず、下地層間絶縁膜（ここでは、シリコン酸化膜）に対するアルミ研磨の選択性が確保されていることが確認できた。ただし、選択性の程度は、セルロース添加による第2の研磨液の方が優れていることも確認された。

【0040】図3は、本発明による第1の研磨液（硝酸アルミニウム添加、濃度 $0.5 \text{ wt} \%$ ）、及び第2の研磨液（セルロース添加、濃度 $4.7 \times 10^{-2} \text{ wt} \%$ ）を用いてAl-Cu合金膜研磨を行った場合の反射率を、研磨前（成膜直後）と比較した図である。なお、図3の反射率の測定は、金属膜の研磨が途中まで進行した段階（すなわち、層間絶縁膜が露出していない時点）で行った。図3より、高温スパッタ成膜直後のAl-Cu膜表面に比べて、いずれの研磨液を用いた場合でも最大7%程度反射率が大きい。これは、研磨によりAl-Cu膜の表面平坦性が向上したためである。硝酸アルミニウム添加とセルロース添加を比較すると、 $300 \text{ nm}$ 付近ではセルロース添加中性シリカ研磨液を用いたアルミ表面の反射率が硝酸アルミ添加中性研磨液に比べて10%以上反射率が大きく、研磨後のアルミ表面の平滑性により優れていることがわかった。なお、セルロース濃度を変化させた場合でも、 $10^{-6} \sim 10^{-1} \text{ wt} \%$ の実験範囲内では、研磨面の反射率に変化はなかった。

【0041】このように、硝酸アルミ添加中性シリカ研磨液およびセルロース添加中性シリカ研磨液には、従来の研磨液に添加されていた過酸化水素水等の酸化剤を添加していないことから、研磨中に金属表面に金属酸化膜や金属水酸化膜が形成されることはない。また、研磨液が中性であることから金属膜が研磨液中に溶解することはないのである。さらに、微少な球形研磨剤粒子を用いることで、金属膜表面の腐食を抑制しているのでは

る。

【0042】なお、第1の発明に関連し、本実施例の研磨対象金属は純アルミニウムではなく $0.5 \%$ の銅を含有したAl-Cu合金であったが、第1の研磨液に添加する電解質塩としては硝酸アルミニウムのみを使用した。これでも十分な研磨特性の安定性が得られた理由は、合金中の銅の含有量が非常に小さいことによる。なお、合金中の主成分以外の添加金属の含有量が $5 \%$ を超える場合には、その添加金属を含有する電解質塩も合わせて添加して研磨液を調製することが望ましい。もちろん、合金中の添加成分金属の含有量が $5 \%$ 以下の微量の場合でも、その電解質塩を添加して研磨液を調製してもよいことは言うまでもない。

【0043】また、本実施例で研磨対象としたような埋め込み配線構造においては、層間絶縁膜と金属配線との間に、バリアメタル層（例えば厚さ数 $\text{nm}$ から数 $10 \text{ nm}$ 程度のチタン等）を挿入することがしばしば行われる。しかし、このようなバリアメタル層の厚さは通常非常に薄く、また層間絶縁膜が露出する直前の研磨最終段階でバリアメタル層を研磨することになるため、バリアメタル層の研磨による研磨特性の変動は通常問題とならない。

【0044】

【実施例2】上述の実施例1において、本発明の電解質塩添加研磨液、および有機高分子添加研磨液のいずれを用いても、安定した金属膜の研磨、および層間絶縁膜に対する金属膜の選択研磨が可能であり、研磨後の表面での腐食発生が抑制出来ることが明らかとなった。しかしながら、両研磨液間には研磨特性の違いも存在し、研磨の進行に対する研磨特性の安定性では電解質添加研磨液が勝っており、一方で金属膜表面の腐食の抑制や層間絶縁膜に対する選択性では有機高分子添加研磨液が優れていることも判明した。以上を考慮すると、電解質添加研磨液は研磨初期からの平坦化研磨工程での使用に適している。この工程は、金属膜表面の凹凸を平坦化し、また層間絶縁膜上の金属膜の多くの部分を除去する工程であり、研磨特性の安定性が最も求められる。また有機高分子添加研磨剤は、その後の残留金属を除去し層間絶縁膜を露出する鏡面研磨工程での使用に適している。この工程では、研磨における高選択比、および金属表面への腐食の抑制が強く求められる。

【0045】そこで、実施例2では、第1ステップとして電解質添加研磨液を用いた平坦化研磨を行い、その後第2ステップとして有機高分子添加研磨液を用いた鏡面研磨を行い、さらに引き続き第3ステップとして洗浄工程を行い、以上の一連の工程により埋め込み金属配線を形成した例を説明する。

【0046】本実施例で使用した研磨装置の概略を図4に示す。本実施例における研磨工程は、上述の通り3つのステップを有している。本実施例の研磨装置は、この

各ステップに対応した3つの研磨ステージを有しており、第1研磨ステージ10では金属膜の概略を除去する平坦化研磨工程を、第2研磨ステージ20では層間絶縁膜を露出すると同時に金属膜表面の仕上げを行う高選択鏡面研磨工程を、第3研磨ステージ30では基板洗浄工程を行うことができる。各研磨ステージは基本的に同一の構成からなり、それぞれ上面に研磨パッドを備えた研磨定盤11、21、31、研磨ヘッド12、22、32、荷重印加機構13、23、33、研磨液供給機構14、24、34を有する。研磨定盤および研磨ヘッドは、それぞれ回転機構により任意の速度で回転させることができ、またその際研磨ヘッドには、荷重印加機構により任意の荷重をかけることができる。研磨対象となる基板15、25、35は表面を下向きとして研磨ヘッドに保持され、研磨液供給機構から研磨液16、26、36を供給することにより基板の研磨または洗浄を行う。なお、第3研磨ステージにおける工程の主目的は基板の洗浄であるが、ここでは研磨液との表現を用いている。

【0047】本実施例で使用した研磨装置においては、以上の第1～第3研磨ステージは、ひとつの支持台1上に配置され、一体構成となっている。また、研磨前の基板はまずローダー2に設置され、その後図示しない自動搬送機構により順次第1～第3研磨ステージに搬送されて3段階の研磨工程を行い、研磨・洗浄終了後の基板がアンローダー3に格納される。以下、各ステージ上の研磨位置にある基板をそれぞれ基板15、25、35として区別する。

【0048】研磨対象とした基板は、実施例1で使用した基板とほぼ同一である。すなわち、あらかじめ層間絶縁膜に深さ500nmの配線溝を形成しておき、その後基板温度450℃の高温スパッタ法でAl-Cu0.5%（厚さ700nm）を埋め込み成膜したものである。Al-Cu合金の成膜条件も、実施例1と同様である。このAl-Cu合金膜上部には、下地配線溝の段差を反映した300nm程度の表面凹凸がある。

【0049】以下、図4における第1、第2、第3研磨ステージでの研磨工程について説明する。

【0050】第1研磨ステージにおける研磨工程は、Al-Cu合金膜表面の表面凹凸を除去する平坦化研磨工程である。ここで用いた研磨液16は、研磨剤粒子として湿式方による粒径40～80nm球形シリカ微粒子を純水に分散させ、さらに硝酸アルミニウムを0.5wt%添加した電解質添加研磨液である。シリカ粒子の濃度は10～20wt%とした。研磨パッドには、硬度90～100程度（JIS-A規格、以下同様）の硬質研磨パッドを用い、荷重印加機構13により0.3～0.4kg/cm<sup>2</sup>の高研磨圧を印加した。研磨定盤11および研磨ヘッド12の回転数は、30～90rpmとした。本工程は、多量のAl-Cu合金膜を研磨除去する工程であり、研磨の高速性が非常に重要である。このた

めに、上記のようにやや粒径の大きい研磨粒子・硬質パッドを使用し、また高圧印加・高速回転の条件下で研磨を行っている。以上の構成により、アルミ残膜が100nmになるまで研磨した。

【0051】第2研磨ステージにおける研磨工程は、下地層間絶縁膜の研磨を極力抑制して選択的にアルミ膜を鏡面研磨する、高選択鏡面研磨工程である。本工程の研磨液26としては、10nm～40nmの球形シリカ微粒子を10～20wt%分散させた純水に、セルロースを0.1wt%添加した有機高分子添加研磨液を用い、軟質パッド（硬度60～90）の研磨パッドで下地層間絶縁膜が現れるまでAl-Cu合金を鏡面研磨した。この時の、荷重印加機構23により印加する圧力は0.3kg/cm<sup>2</sup>以下の低圧力とし、また研磨定盤21、研磨ヘッド22の回転数はそれぞれ10～40rpmとした。ここで、粒径の小さい粒子・軟質パッドを使用し、また低圧・低速回転の条件下で研磨を行うのは、研磨後のAl-Cu合金膜における研磨面の平滑性を向上させるため、および研磨の選択性をより高くするためである。本工程により、表面凹凸のない埋め込みアルミニウム配線（ダマシナーアルミニウム配線）が得られる。

【0052】第3ステージにおける研磨工程は、第2ステージまでの研磨工程により形成された、Al-Cu膜と層間絶縁膜からなる複合表面を洗浄する工程である。本工程で使用する研磨液36としては純水を使用した。また、研磨パッドとしては硬度60以下の軟質パッドを使用し、荷重印加機構33による印加圧力は0.3kg/cm<sup>2</sup>以下、研磨定盤31、研磨ヘッドの回転数40rpm以下の条件で研磨を行った。なお、本工程で用いる研磨液36としては、純水に0.1wt%以下のセルロース等の有機高分子（界面活性剤）を添加したものを用いてもよい。

【0053】以上の一連の研磨工程で、層間絶縁膜に厚さ500nmのAl-Cu合金が埋め込まれたダマシン配線を形成した。このAl-Cu配線表面の凹凸は10nm以下であり、平滑性に秀でていることが確認された。

【0054】本実施例においては、図4に示すように第1から第3の研磨ステージ接続し、基板をステージ間で自動搬送できるような研磨装置を使用した。しかし、個別の研磨装置を複数用意して、それぞれに第1、第2、第3の研磨工程を行わせるようにしても本発明の効果は全く同様である。また、以上の3工程の内でも本質的に重要な工程は第1および第2の研磨工程であり、第3の洗浄工程は別の洗浄手段によっても良い。

【0055】

【発明の効果】従来の金属研磨においては、微細研磨剤粒子の分散液にpH調整剤や酸化剤を添加した研磨液を用いていたため、金属表面に表面絶縁膜が形成されたり、研磨により金属表面に凹凸が発生する、さらに研磨

の進行とともに研磨特性が変動するなどの技術課題があった。

【0056】しかし、本発明によれば、中性研磨液に研磨対象の金属膜を構成する金属元素を含む電解質塩あるいはセルロース等の有機高分子を微量添加した研磨液を使用することにより、上記課題を解決することが出来た。すなわち、あらかじめ凝集させた研磨剤粒子で直接金属面を研磨することで平滑性に優れた金属研磨を達成し、また研磨に伴う研磨液の特性変化を防止することにより研磨特性の安定が得られた。

【0057】また、埋め込み金属配線の形成においては、第1の研磨工程として電解質塩添加研磨剤を用いた平坦化研磨を行い、第2の研磨工程として有機高分子添加研磨液を用いた高選択鏡面研磨工程を行うことにより、表面凹凸のない埋め込み配線の形成を行うことが可能になった。

【0058】以上の結果、安定したダマシ配線の形成が可能となり、配線信頼性が著しく向上するという顕著な効果が得られた。

#### 【図面の簡単な説明】

【図1】本発明の研磨液によるAl-Cu合金膜の研磨速度と研磨圧力の関係を示す図である。

【図2】本発明の第2の研磨液中の有機高分子濃度とAl-Cu合金膜の研磨速度の関係を示す図である。

【図3】本発明に研磨液により研磨したAl-Cu合金の反射率を示す図である。

【図4】本発明による埋め込み配線形成研磨方法を示す工程断面図である。

【図5】従来の研磨装置を示す図である。

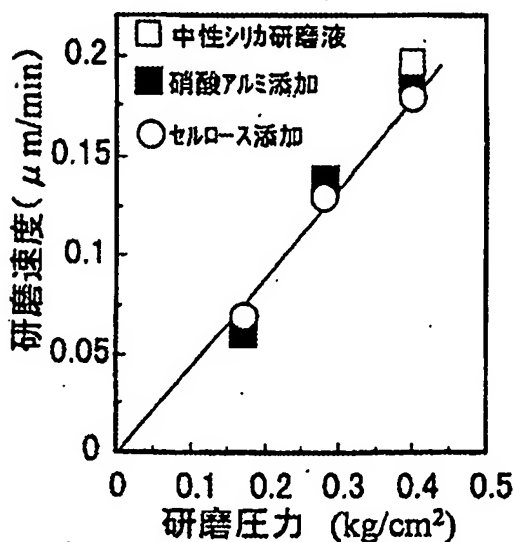
【図6】従来の埋め込み配線形成工程を示す工程断面図である。

【図7】従来の研磨方法における研磨ヘッドトルクの時間依存性を示す図である。

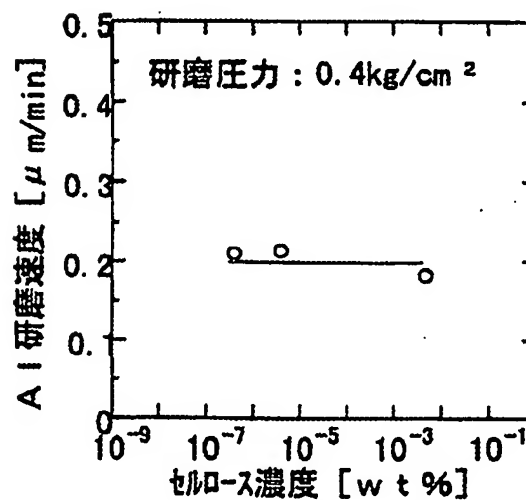
#### 【符号の説明】

- 1 支持台
- 2 ローダー
- 3 アンローダー
- 10 第1研磨ステージ
- 20 第2研磨ステージ
- 30 第3研磨ステージ
- 11、21、31 研磨定盤
- 12、22、32 研磨ヘッド
- 13、23、33 荷重印加機構
- 14、24、34 研磨液供給機構
- 15、25、35 基板
- 16、26、36 研磨液
- 61 基板
- 62 下層配線
- 63 層間絶縁膜
- 64 ピアホール
- 65 金属膜

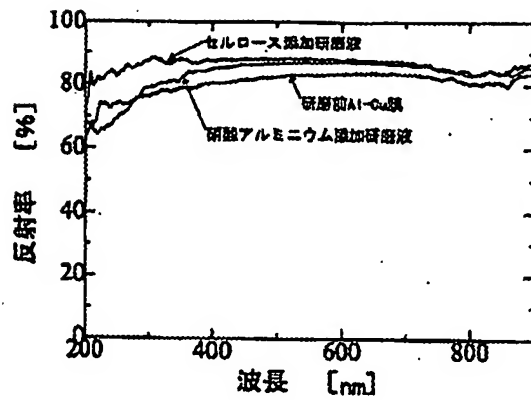
【図1】



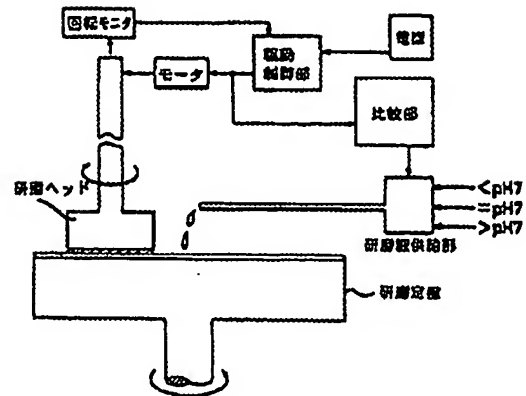
【図2】



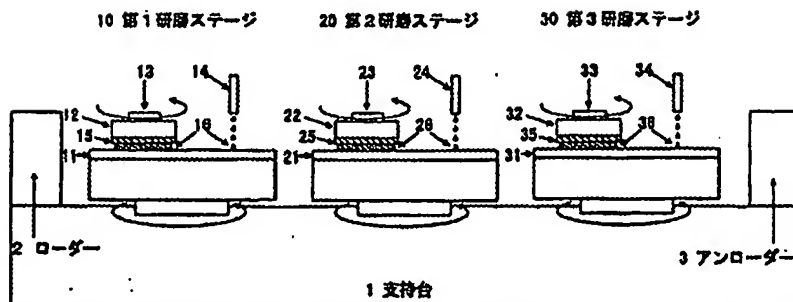
【図 3】



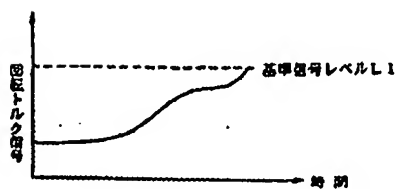
【図 5】



【図 4】



【図 7】





【図6】

